ENERGY CONSERVATION BY CHEMICAL COMMINUTION OF ASBESTOS

FINAL REPORT

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Chemical Comminution of Asbestos
Final Report

EXECUTIVE SUMMARY

To date, all processing of asbestos ore is achieved by means of dry techniques which employ mechanical crushing, screening, etc. to separate fibre from host rock and to classify the fibre on the basis of length distribution. This method of processing requires drying of the crushed ore prior to milling, as well as the movement of large volumes of air to separate, transport and collect fibre and to comply with occupational health requirements. Obviously, the dry processing of asbestos ore is very energy intensive.

An alternative to the dry process is wet milling, replacing air by water as the medium of separation. This may involve the use of chemicals such as surface active agents during the comminution step as demonstrated by the process developed recently in Australia. It is claimed that wet processing of asbestos ore has a number of benefits such as improved fibre recovery.

A comparison of the major processing stages employed in both the conventional dry lifting operation and the wet milling process are illustrated below.

	Dry Process	Wet Process
Primary Crushing	x	x
Ore Drying	x	
Comminution	x	x
Air Movement	x	
Water Recycling		x
Fibre Dewatering		x
Tailings Disposal	x	x

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A study has been carried out to compare only the comminution steps of both processes with regard to energy consumption, fibre yield and quality by processing a Canadian ore on a small batch pilot plant scale. Operational parameters such as concentration and type of surfactant as well as different comminution or separation techniques have also been examined. The fibres produced by these processing methods have been characterized by conventional test methods known in the asbestos industry as well as by a variety of analytical techniques.

Based on these studies, the following conclusions are presented.

(1) Significant energy savings are observed for wet comminution of asbestos as compared to dry comminution.

(2) Fibre yield is increased significantly by employing ballmilling with a surfactant.

(3) Ballmilling tends to shorten fibre (i.e. reduce the length).

(4) The use of surface active agents may have a detrimental effect on fibre properties.

(5) Multiple fibre grades may be produced by wet screening or classification.

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1. INTRODUCTION

Conventional dry milling is the only process currently employed by producers of cement or spinning grade asbestos fibres. This process involves a series of successive steps of crushing or grinding of ore to release and separate the fibre from the host rock by screening and air aspiration. Dry milling is an energy intensive process, not only because of the use of mechanical systems, but also because large amounts of air must be circulated to aspirate and collect the fibre.

Also, to meet occupational health requirements, it is necessary to install additional baghouse units to control dust and recirculate filtered air. A typical mill will require 12 - 15 tonnes of air (10,000 to 12,500 m^3) per ton of ore milled, with more than half being employed for dust control.(1) In addition, the ore must be dried prior to processing to allow efficient recovery of fibre.

When one considers that in 1982 the ten Canadian mines produced 822,000 tonnes of fibre (25% of world production), it becomes obvious that the required energy is enormous. In 1978, the total Canadian energy consumption associated with the processing of asbestos ore was estimated to be in excess of 10^{16} Joules.(2)

There is a current worldwide trend to control asbestos fibre dust levels in the workplace to values as low as 0.5 fibre/mL of air. Such values may be difficult to maintain when using dry milled fibre. The implementation of a wet process may therefore solve this occupational health problem.

Wet processing of asbestos saw limited use in one Canadian plant in the early 1950's(3). The primary goal was to recover short fibres from mill tailings to produce marketable commercial grades. The process,

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however, became very energy intensive by requiring the fibre product to be dried to approximately 1% moisture for commercial applications. A later study examined pilot plant recovery of asbestos fibre from plant tailings using the wet processing method[4].

Union Carbide has been operating a wet milling operation with an unusual asbestos ore in California to produce fillers and fibres for special applications. More recently however, an alternative wet milling process using a wet chemical comminution technique for the recovery and grading of asbestos fibre from ore has been developed in Australia by Woodsreef and I.C.I. This technology is similar to the chemical techniques used in the manufacture of asbestos yarn from dry-milled fibre. When applied to ore, a dilute aqueous solution of a surface active agent separates fibre from the host rock. Australian investigations have also shown that the yield of useful asbestos fibre per tonne of ore is increased considerably. However, little information exists on the properties of the asbestos recovered by this technique.

A variation of this process using no chemicals has been developed to the prototype mill stage. Extensive plant trials have shown that the wet milled fibre produced performs well during asbestos-cement production without creating occupational health problems.

The following report describes work carried out in an attempt to define the expected energy requirements involved in the wet comminution of ore during wet milling in comparison to the conventional dry milling process. In an effort to characterize the fibre recovered by the wet milling techniques, an investigation into the structure and properties of the individual wet processed fibres in comparison to the dry milled fibres has been conducted.

2. LITERATURE REVIEW AND PROCESS ASSESSMENT

2.1 Wet Milling of Asbestos

In recent years, interest in wet milling processes as an alternative to conventional dry milling for the separation of asbestos fibre from its ore has been increasing. It is generally considered that the wet process has one or more of the following benefits:

- o improved recoveries of product;
- o reduced environmental and occupational health hazards;
- o reduced energy costs to process.

However, both the technological and capital investment by producers in conventional dry milling procedures is extremely high and due to the competitive market, they are hesitant to launch an 'unknown' product. As a result, although interest in a wet milling process exists, none of the major Canadian manufacturers has progressed beyond test work and/or small pilot scale production. Thus, there is a requirement to review and assess work conducted in this area to date and the quality of product produced, and to analyze the possibility of developing a viable wet milling process for asbestos production. As part of this program, such a review has been undertaken.

2.1.1 Wet Mill Steps

The wet milling of asbestos may be conveniently divided into the following stages:

- o fibre liberation and separation;
- o fibre opening and grading;
- o fibre dewatering;
- o water recycling and tailings disposal;
- o packaging.

A brief discussion of these stages is presented below.

2.1.1.1 Fibre Liberation and Separation

Wet milling of asbestos dates back to the 1920's when several patents[5-12] were issued for the wet separation of asbestos fibres from their gangue. These patents were mainly concerned with the wet grinding and crushing of asbestos bearing rocks and the use of water as the vehicle to separate asbestos from gangue material. This separation was achieved principally by flotation and sedimentation processes.

More recently [13, 14, 15, 16] conventional wet mineral processing techniques have been applied to effect a separation; these techniques include crushing, grinding, sedimentation, tabling, thickening filtration, and hydrocycloning.

Other techniques [17, 18] include heating an asbestos slurry under high pressure followed by blasting to release the fibre and heavy media separations, principally to remove magnetite and similar impurities.

Thus, it would appear that the separation of asbestos fibre from the host rock by wet methods is feasible and practicable. Modern practice would require the incorporation of closed-circuit milling to minimize water usage and simplify recycling and tailings disposal.

Such a system should also minimize energy usage. Closed circuit wet milling can be achieved by the use of standard crushers and fibrizers in conjunction with wet separators. Typical wet separators are settling basins, wet shaking tables, rake and screw classifiers. Rake and screw classifier separators are particularly effective in the separation of coarse grains owing to their counter-current action. Examples are the Hardinge (screw) classifier and the Dorr-Oliver rake classifier. In recent years, however, hydrocyclones have become the most popular wet separation technique owing to the compact design and economy of operation. Wet screening has also been used but operating costs are sometimes high due to measures required to prevent screen blinding.

2.1.1.2 Fibre Opening and Grading

Asbestos fibre opening may be accomplished by a combination of chemical treatment [19] and mechanical processing [20, 21]. Chemical treatment includes soaking in aqueous solutions containing dispersing agents, surfactants, acids and alkalis. Mechanical processing may be carried out using ball and rod mills as well as high-sheer agitators. By judicious choice of chemical agents and mechanical processing, a high degree of fibrization can be achieved. The use of chemical to enhance fibre opening will be discussed in Section 2.2.

Wet grading of fibres is problematic but it is potentially workable. For example, wet screening is plagued with problems due to the blinding of the screen openings. However, the use of rubber screens and mechanical vibration may overcome this problem. Hydrocyclones may have some potential for the grading of asbestos fibres and other wet classifiers such as elutriating cones, bowl classifiers, rake and spiral classifiers and solid-bowl centrifuges may also be suitable.

Parratt [22] describes a patented length-grading apparatus for the continuous classification of asbestos fibre slurries. This machine operates on the principle of feeding the asbestos suspension onto a rotating cylindrical screen at a rate that permits only a single layer of fibres to be retained while the shorter fibres pass through the mesh via centrifugal action. This equipment is described more fully by Gooding et al [23].

2.1.1.3 Fibre Dewatering

Dewatering of an asbestos slurry may be achieved by a combination of thickeners, filters and presses to a water content of about 15%. Any further drying of the filter cake which involves the application of elevated temperatures, is energy intensive and, will therefore add significantly to the overall cost of the wet milling process. However, it seems likely that asbestos-cement producers would be willing to accept a wet raw material. Thus the above drying step could be avoided at significant savings to the asbestos producers, although the residual water content may add to the shipping cost.

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2.1.1.4 Water Recycling and Tailings Disposal

Coarse tailing particles present no particular problem. These may be removed by standard techniques such as the hydrocyclone. Fine tailing particles are best removed by the use of flocculating agents followed by filtration. However, it is recognized that the additions of more chemicals to the system should be avoided if at all possible for the purpose of water recycling. In addition, recycled chemicals may have a detrimental effect on the quality of the asbestos fibres. An alternative to this approach is to pond the tailings materials.

2.1.1.5 Packaging

Conventional dry milled fibre is "pressure packed" and sold in 50 kg plastic bags at a bulk density of about 880 kg/m^3 (). Wet processed fibre is likely to contain at least 15% water, thereby adding a weight to volume penalty to such a packaged product. Since conventional drying techniques are extremely energy intensive, it appears logical that the product should be dewatered by mechanical means as much as possible, and packaged and shipped "wet" presumably in the form of a felt or pellets. It has been shown that such felts and pellets disperse readily in asbestos-cement production with less environmental and occupational hazards than the current dry processed fibre.

2.1.2 Advantages and Disadvantages of Wet Milling

Wet milling of asbestos offers several advantages over conventional dry milling. From an occupational health standpoint, the most obvious advantage is the virtual elimination of carcinogenic asbestos dust in the workplace (e.g. asbestos-cement plants). From a milling standpoint, wet milling results in the preservation of fibre length and a probable energy benefit. In wet milling, the addition of suitable chemical agents increases grinding rates by weakening fibre adhesion along the cleavage planes, by preventing particle agglomeration and by preventing ball coating in ball mills. Moreover, the strength of porous rocks is significantly lowered by water, thus, contributing to some savings in energy.

A further advantage of wet milling with or without chemicals, as shown by the Australian studies, is the recovery of significant fibre quantities from previously discarded tailings from dry mill operations. It seems likely that, at the outset, this may provide the initial impetus for the commercial application of a wet mill.

Among the disadvantages of wet milling is the cost of drying if this should be required. Filtration as a dewatering process results in a product with a high (~60%) water content. Other dewatering techniques may be employed (e.g. centrifuging) to reduce the water content to about 15%. Further drying is energy and hence cost intensive. As wet processing in the manufacturing of asbestos products becomes a common practice, an alternative approach may be to persuade asbestos-product manufacturers to accept a partially-dried material. However, this energy saving may be offset by the added cost of shipping water along with the material.

Chemicals such as dispersants, surfactants, acids and alkalis may be necessary in wet milling. However, the quality of fibres for specific applications may be detrimentally affected by the use of chemical additives, particularly if the additive is retained or adsorbed on the fibre surface after processing. Therefore, the nature and concentration of these chemical additives have to be carefully examined and techniques to ensure their removal at the end of the milling process need to be implemented and/or developed.

2.2 Chemical Comminution

2.2.1 Historical Background

Early work involved with the processing of chrysotile asbestos with water began as early as 1920 when Selective Treatment Company Ltd. conducted laboratory research into a wet method of treating asbestos ores from Thetford Mines, Quebec [24]. Following this investigation, an experimental pilot plant was erected in 1923 and although sufficient work was completed to demonstrate that the process provided increased yields as compared to dry methods, a commercial plant was never constructed.

In 1922, Carnochan [25] reported work conducted at the Ore Dressing and Metallurgical Division of the Canadian Mines Branch. This work was somewhat inconclusive as to selecting the optimum method (wet or dry) but suggested further study on a larger scale. The following year, Mett [26] patented a closed loop system for refining asbestos ore, and in 1929, Carnochan [27] published results which showed simple wet processing with water to be economically viable at that time.

Some of the premier work involving the addition of chemical agents began in 1931 when Tucker [28] described the dispersion of opened asbestos fibres in aqueous media by means of mechanical action and the addition of chemical agents such as aluminum chloride or thorium nitrate. He observed a "visible increase in the dispersive dissemination" of the asbestos fibres using this procedure.

The use of chemical surfactants was not employed until the 1950's when Novak [29] described a variety of possible surfactants and hypothesized on their effect with regard to the formulation of asbestos dispersions. Although Novak was primarily interested in the preparation of asbestos/paper sheets, he did examine the effects of surfactant concentration and temperature on the degree of openness obtained. He also commented on the proportion of free wetting agent that remains on the fibre.

Barbaras [30] also studied the use of chemicals in the formation of aqueous asbestos dispersions. His work, however, dealt primarily with the use of dissolved metal salts having a monovalent anion and a cation containing a polyvalent metal. In that same decade, Rex Asbestwerke [31] was awarded a German patent dealing with the manufacture of asbestos yarn from asbestos dispersions containing a fatty acid soap.

In 1971, Rex Asbestwerke [32] was awarded an American patent for work dealing with the dispersion of chrysotile asbestos in aqueous media with an anionic soap as a dispersing medium in conjunction with the use of a "sharpening" agent such as aluminum sulphate which aided in the coagulation of the fibre slurry to form asbestos yarn. This addition was found to improve the storability and extrudability of the dispersion as well as the tensile strength of the manufactured asbestos strands.

Other important works dealing with the use and/or treatment of chemically comminuted fibre include three patents by Fetzer [33], Schultz and Fogel [34], and Xanthos and Woodhams [35]. The former patent deals with the removal of the residual surfactant from the treated fibre by extraction with low aliphatic alcohols and/or aqueous solutions of organic bases. Fetzer also discusses a number of previous studies which examine such removal techniques as heating, washing, solvent extraction, and steam heating.

Schultz and Fogel examined the formation of asbestos fibre dispersions in aqueous slurries containing a specified concentration of a selected acrylic or methacrylic acid polymers.

Xanthos and Woodhams investigated the chemical opening of chrysotile asbestos into the individual fibrils by the use of soluble vinylic polymer polyelectrolytes. These individual fibrils were then encapsulated by co-polymerization to form a variety of reinforced composites including laminates and foamed or cellular products.

In 1976, two German patents were awarded to TBA Industrial Products Ltd. of England [36, 37] which dealt with the formation of asbestos fibre dispersions. The former work describes a method for the preparation of asbestos fibre dispersions in an organic hydrocarbon liquid (e.g. benzene, toluene, mineral spirts, chloroform) with the

addition of an alkylarylsulphonic acid which acts as a wetting agent. The fibres were opened by this process until an average aspect ratio of 10^3 had been reached. The latter patent is more concerned with the production of asbestos yarn and thread from aqueous chrysotile fibre dispersions formed by the addition of excess fatty acid soap.

The use of surfactants containing solutions to recover asbestos fibre in the mining process has been studied extensively in Australia. Two recent patents by Absolon et al [38, 39] discuss both the actual process of obtaining chemically comminuted fibre and its use in asbestos cement composites. A wide variety of possible surfactants are examined and their suitability to the wet comminution process evaluated. This technique of refining asbestos ore is claimed to increase the overall yield of asbestos-cement grade fibre from the 2 - 6% weight by weight yield recovered in the conventional dry process to 8 - 15% weight by weight using the chemical comminution process. This does not necessarily imply that the overall quantity of asbestos-cement grade fibre collected is increased that dramatically, rather that a feature of this process is the production of an elongated agglomerated fibre capable of imparting good strength in cementitious composites, from previously unacceptable low grade short fibre.

Kharsas et al [40] also describe a chemical method involved in the preparation of asbestos fibres which is very similar to that of Absolon et al [39]. This work, however, examines the use of a closed loop system by employing ultrafiltration to remove and/or concentrate the surfactant from the process stream. The permeate solution can thereby be treated and employed as a source of recyclable aqueous medium.

2.2.2 Surfactants

A very broad range of surfactants have been reported [38, 39, 40] as being useful in separating asbestos fibre from host rock. They are selected from the classes of anionic, cationic, nonionic, and amphoteric

surfactants. A surfactant solution can be prepared by mixing surfactants of the same class or by mixing nonionic with anionic surfactants. Nevertheless, mixing surfactants is not a preferred method of operating because it complicates aspects of reagent recycling. The preferred method is to use anionic surfactants, particularly dioctyl ester of sodium sulphosuccinate (Aerosol OT) either alone or with a small proportion of dodecylbenzene sulphonic acid (DBSA).

3. EXPLORATORY BENCH SCALE STUDIES WITH FREE FIBRES

3.1 Preliminary Survey

A wide variety of possible surfactants for use in the chemical comminution of asbestos have been listed by Novak [29] and others. These include anionic surfactants (e.g. carboxylates, sulphates, sulphonates, and phosphates), cationic surfactants (e.g. amines, amine oxides, and quarternary ammonium salts), and non-ionic surfactants (natural fats and oils, alkoxylated aliphatic alcohols, and fatty acids). The selection of a suitable surfactant will depend on its overall effectiveness (i.e. ease of handling, degree of opening attained, removal and recovery) with respect to a particular fibre type and, of course, its availability, cost and toxicity.

For the purposes of this study, three anionic surfactants were chosen based upon previous literature surveys and research conducted at Ontario Research. These surfactants were also readily available and posed few problems with regard to handling. The two carboxylates chosen were sodium oleate (J.T. Baker Chemical Co.) and sodium laurate (K and K Laboratories Inc.). These two surfactants were shown by Kharsas et al [40] to give good results when applied to the chemical comminution of Australian chrysotile. Aerosol OT-75 (dioctyl ester of sodium sulphosuccinate) (Canadian Cyanamid) was also investigated as it had been shown by Novak[29] to be one of the most effective surfactants used in the wet chemical opening of Canadian chrysotile.

A preliminary investigation of these three surfactants was conducted on a Canadian fibre which had previously been dry processed through the Ontario Research pilot plant (see Section 4.3). A number of 1% weight by weight aqueous slurries of this fibre were prepared. The following surfactant concentrations (based on total slurry weight) were effected on the slurries:

Aerosol OT - 0.01%, 0.05%, 0.1%;
Sodium Oleate - 0.02%, 0.1%, 0.2%;
Sodium Laurate - 0.02%, 0.1% 0.2%;

and then breifly agitated and allowed to stand overnight. The degree of fibrization was then evaluated visually and the fibre was collected by suction filtration. Both of the carboxylates tended to impart a brownish colour to the fibre after drying, the sodium laurate to the extent that it was omitted from further testing. Based on the viscosity of the fibre slurries, it appears that the Aerosol OT had opened the fibres to a greater degree than the other two chemicals.

The control of pH in an aqueous slurry of asbestos may be an important factor in determining the degree of fibrization that may occur. Chrysotile fibre has been shown by Ahmed [41] to have a zero point of charge around pH = 10. Below this value, an acidic dissociation of the Mg (OH) group, and/or a H^+ adsorption is believed to occur which leads to the formation of a positive surface charge. Such a charged surface would be conducive to the attraction of any anionic species present (i.e anionic surfactants). This attraction may therefore be responsible for the splitting of asbestos fibre bundles.

The pH of a few aqueous slurries (0.2 - 1.0% weight by weight fibre) were measured and presented in Table 3.1.

TABLE 3.1

	SURFACTANT		
Concentration	Aerosol OT	Sodium Oleate	Sodium Laurate
0.2%	7.4	8.6	8.3
0.5%	8.5	9.5	9.6
1.0%	8.9	9.6	9.9

Based on the above hypothesis, Aerosol OT is expected to be a more suitable surfactant due to its less basic nature, thereby increasing the magnitude of the positive surface charge. Examination of the surface charge density with varying pH will be discussed later in this report (see Section 6.3).

3.2 Contacting Techniques

Two methods were investigated; ball milling, and mechanical stirring. A preliminary ball milling study on slurries of 1% asbestos fibre by weight were conducted. Using five inch I.D. mills and a charge of 240:1 balls to fibre by weight, slurries with two concentrations of Aerosol OT and sodium oleate (0.05% and 0.5% w/w) were run at two milling times (one hour and four hours at 87 rpm) along with aqueous controls at identical conditions.

The high surfactant concentration tests (0.5%) produced extremely coagulated stringy slurries which were very difficult to dewater by suction filtration. The lower concentration slurries (0.05%) were easy to handle and dewatered well but showed evidence that ball milling for an extended period (four hours) tended to shorten the recovered fibre. It was thus proposed that all subsequent ball milling not exceed one hour in duration.

A more extensive ball milling study was conducted on 1% asbestos fibre slurries using fibre dry-lifted from the ore selected for this project. The milling was scaled up by using 9-inch mills with a charge of 220:1 balls to fibre by weight. Three different surfactant concentrations (Aerosol OT - 0.05%, 0.1% and 0.2%, sodium oleate - 0.1%, 0.2% and 0.4%) and three mill times (60, 30 and 15 minutes) were investigated. The same experiments were carried out on controls, i.e. without the use of surfactants.

A similar study involving mechanical mixing of slurries prepared with the same asbestos/surfactant ratio and agitated for 30 and 120 minutes was also completed. It was noted from these two studies that the dried fibre samples which had been treated with sodium oleate were discoloured (brownish) and had a rather stiff character. For these reasons, further testing was conducted on Aerosol OT only.

To assess the degree of fibrization which had occurred in both the ball milling and mechanical mixing processes, B.E.T. (gas adsorption) surface area analysis was employed. To relate the B.E.T. values to those of conventional air permeability techniques (ASTM D 2752, QAMA D3), a correlation curve (Figure 3.1) was prepared by mechanically opening dry milled fibre in a Waring Blendor and measuring the surface area by both methods. The observed values were:

Sample	Air Permeability (cm^2/g)	B.E.T. (m^2/g)
L-1 (see Fig. 4.1)	4500	15.8
L-1 opened 5 sec.	7100	17.0
L-3 (see Fig. 4.1)	10,400	19.9
L-1 opened 23 sec.	14,500	19.3
L-1 opened 95 sec.	20,100	22.1

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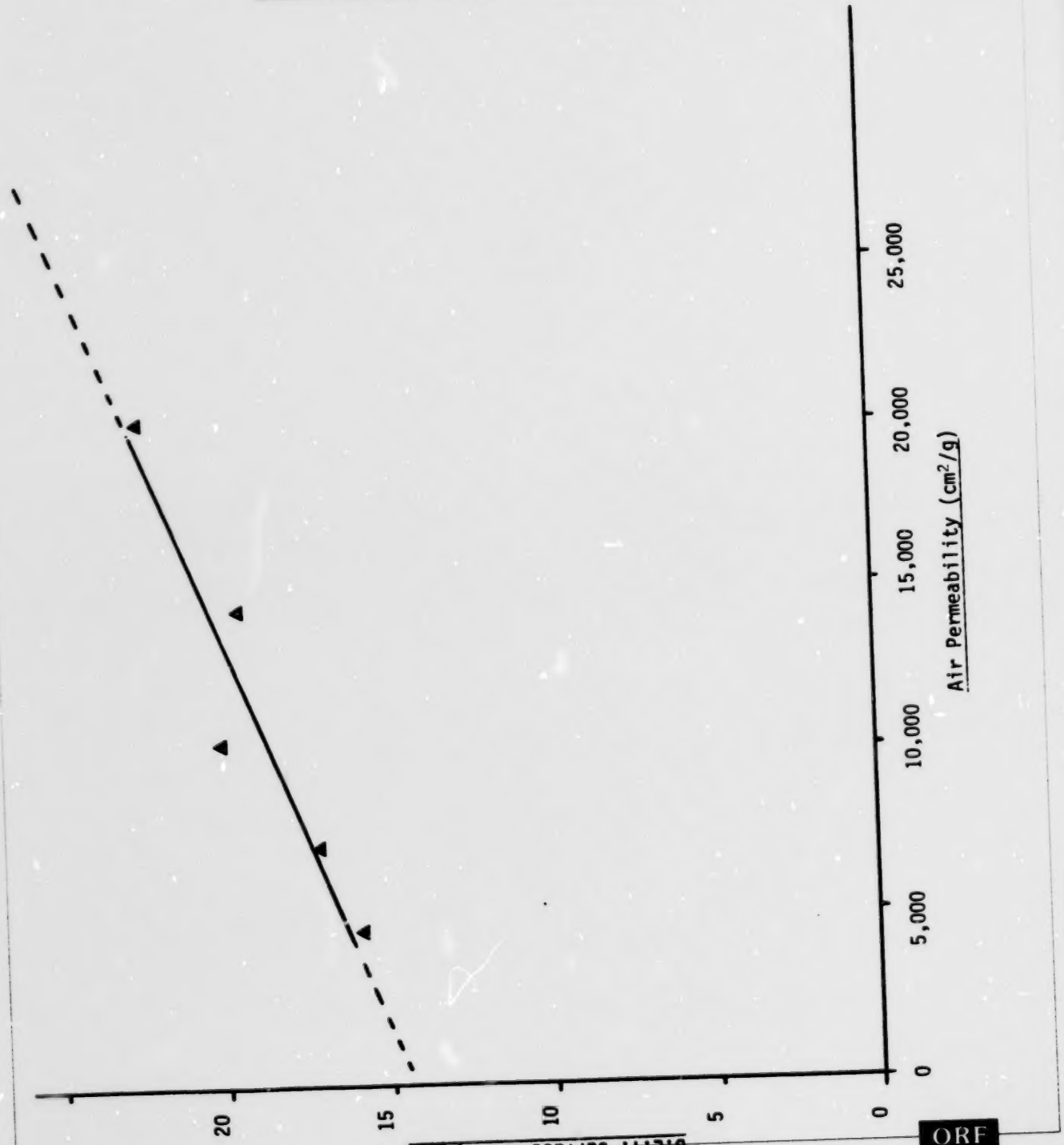
FIGURE 3.1

B.E.T. SURFACE AREA VS AIR PERMEABILITY



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The following samples were contacted and analyzed by the B.E.T. method to assess the degree of fibre opening.

Sample	B.E.T. (m^2/g)	(Air Permeability (from Fig. 3.1) (cm^2/g)
1% fibre w/w ball milled 15 min. in water	22.2	19,400
1% fibre w/w ball milled 30 min. in water	23.0	21,500
1% fibre w/w ball milled 60 min. in water	21.9	18,800
1% fibre w/w stirred 30 mins. in water	21.3	17,400
1% fibre w/w stirred 120 mins. in water	21.9	18,800
1% fibre w/w ball milled 30 mins. in 0.1% Aerosol OT	13.5	--

From these results, it is apparent that chemical comminution may not be needed to open this particular fibre to a level suitable for asbestos product evaluation. Simple mechanical agitation in water may indeed be sufficient to adequately open the fibre. Surface area analysis of the chemically treated fibre shows it to be relatively low. This is probably due to the actual bonding and clogging of fibrils by the residual surfactant. In order to assess the practicality of chemical opening, it is therefore necessary to remove any residual surfactant from the fibre surface.

3.3 Surfactant Removal

Two methods to remove the residual surfactant from filter cakes of chemically comminuted fibre have been attempted. The first technique involves washing or dispersing the treated fibres in a caustic solution (NaOH) or suitable solvent (methanol). The second procedure involves heat treating the sample to actually "burn off" any residual organics.

In this study, differential thermal analysis (DTA) has been used to evaluate the degree of surfactant removal. Figure 3.2 shows a differential thermogram for the plain dry-lifted Canadian fibre. The small endothermic peak around 360°C is thought to arise from the dehydroxylation of unbound brucite ($Mg(OH)_2$) present in small amounts with the asbestos fibre.

Figure 3.3 shows the differential thermogram for the fibre milled for 30 minutes with 0.1% Aerosol OT. The presence of adsorbed surfactant is primarily observed by the large exothermic peak around 300°C.

To investigate the effects of washing the treated fibres, DTA's were run on:

(a) fibres washed in 1M NaOH, (Figure 3.4)
(b) fibres washed in methanol, (Figure 3.5) and,
(c) fibres washed twice in methanol (Figure 3.6)

Of these three treatments, the double washing in methanol appears to remove the majority of adsorbed surfactant; however, a significant amount can still be observed on the fibre as evidenced by the distinct exotherm around 275°C.

FIGURE 3.2

DTA THERMOGRAM OF DRY-LIFTED FIBRE

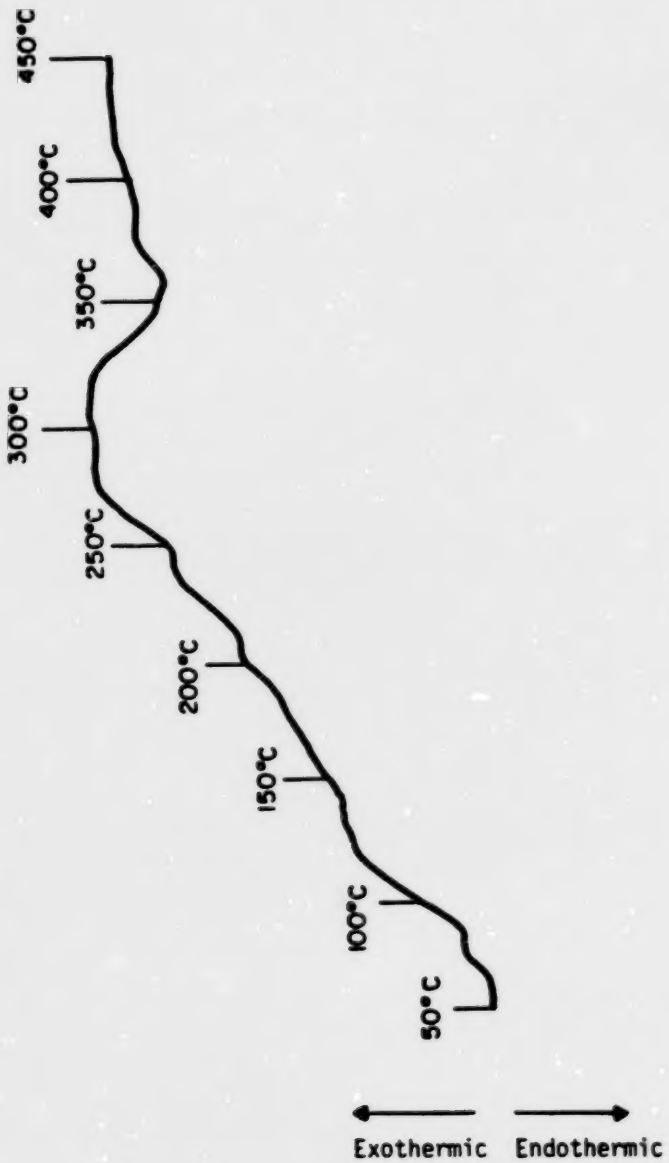


FIGURE 3.3

DTA THERMOGRAM OF 0.1% AEROSOL OT FIBRE

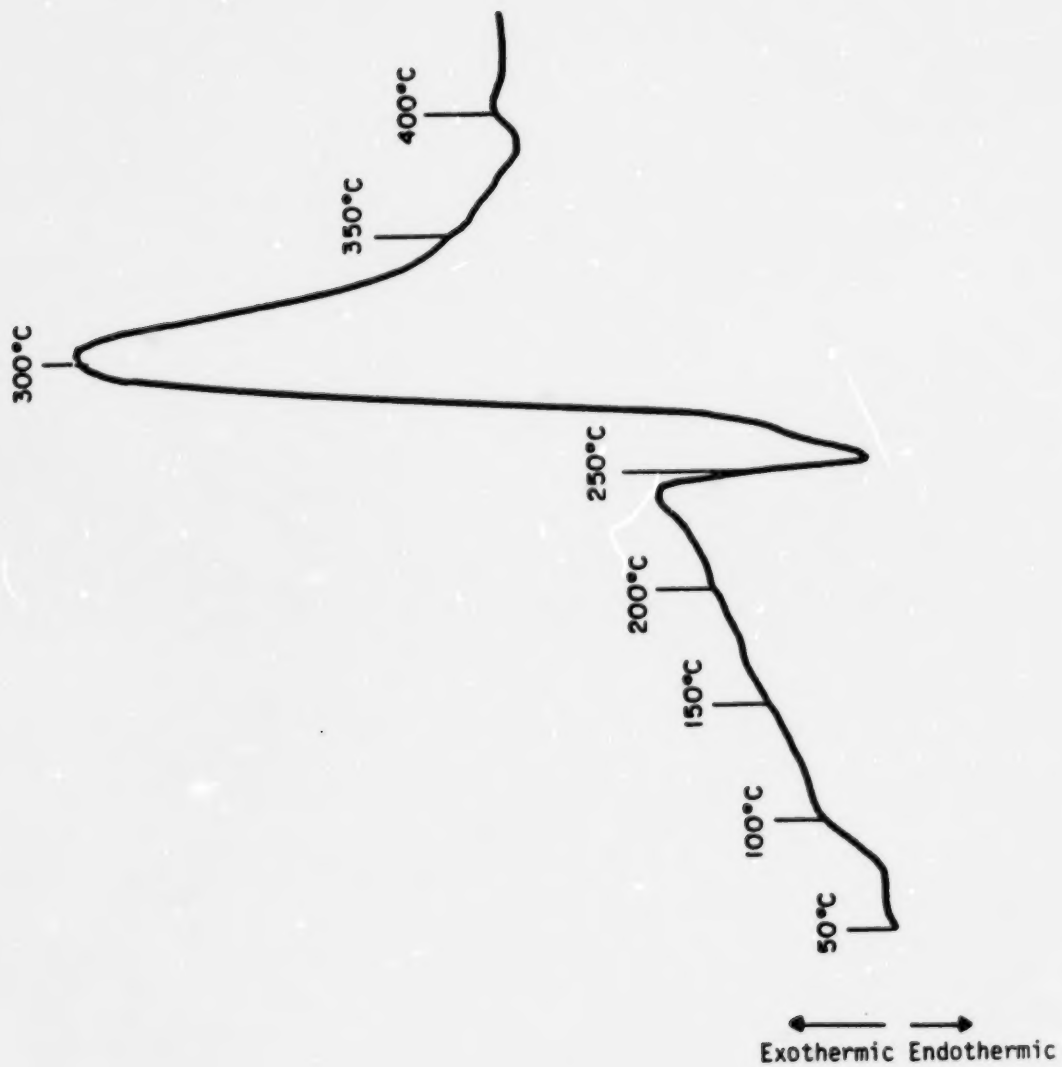


FIGURE 3.4

DTA THERMOGRAM OF 1M NaOH WASHED FIBRE

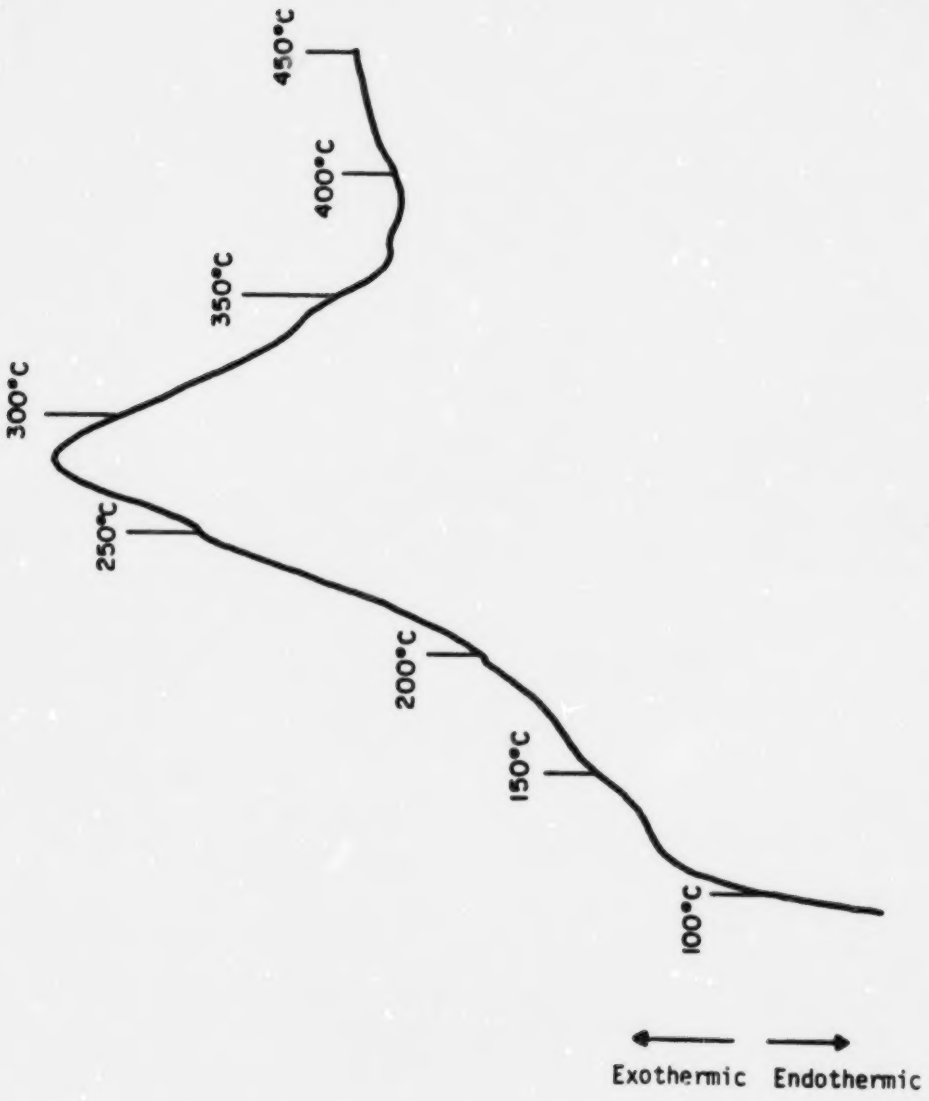


FIGURE 3.5

DTA THERMOGRAM OF METHANOL WASHED FIBRE

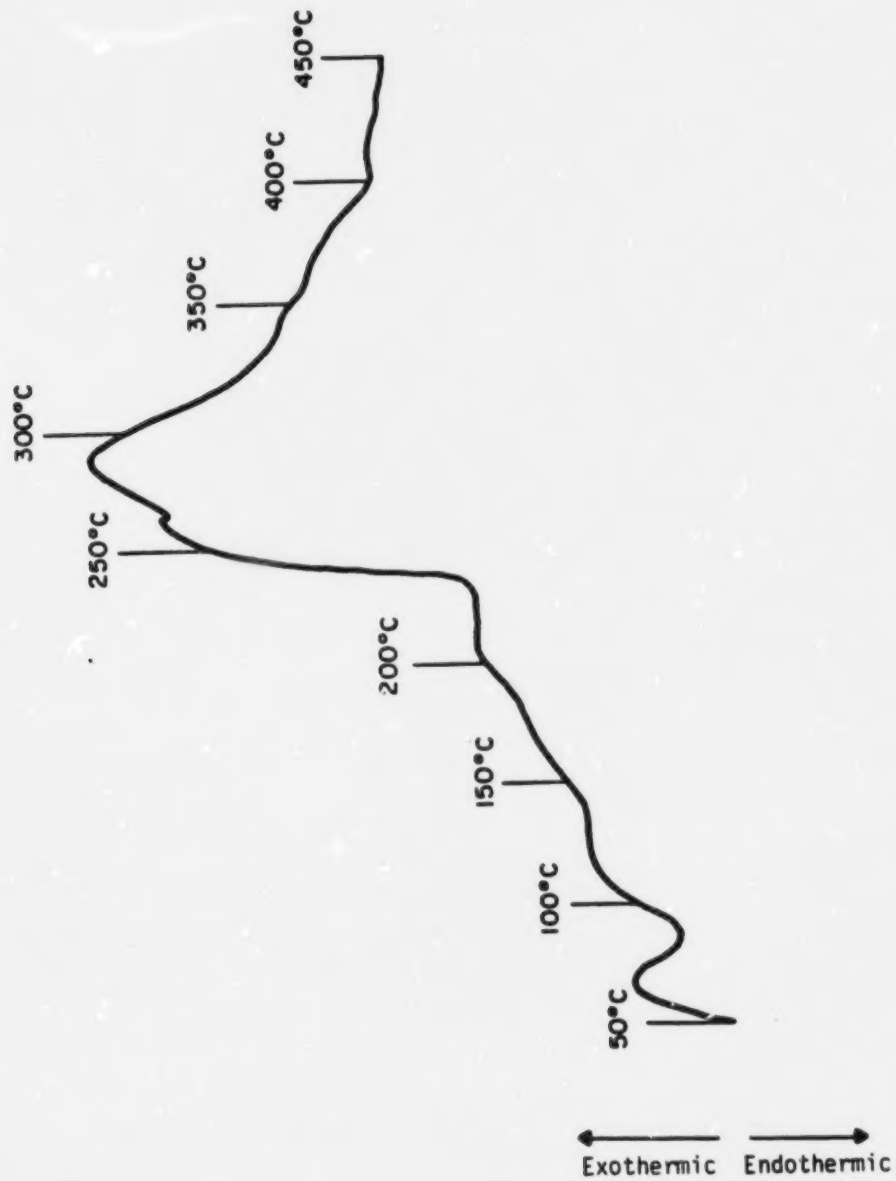


FIGURE 3.6

DTA THERMOGRAM OF METHANOL DOUBLE WASHED FIBRE

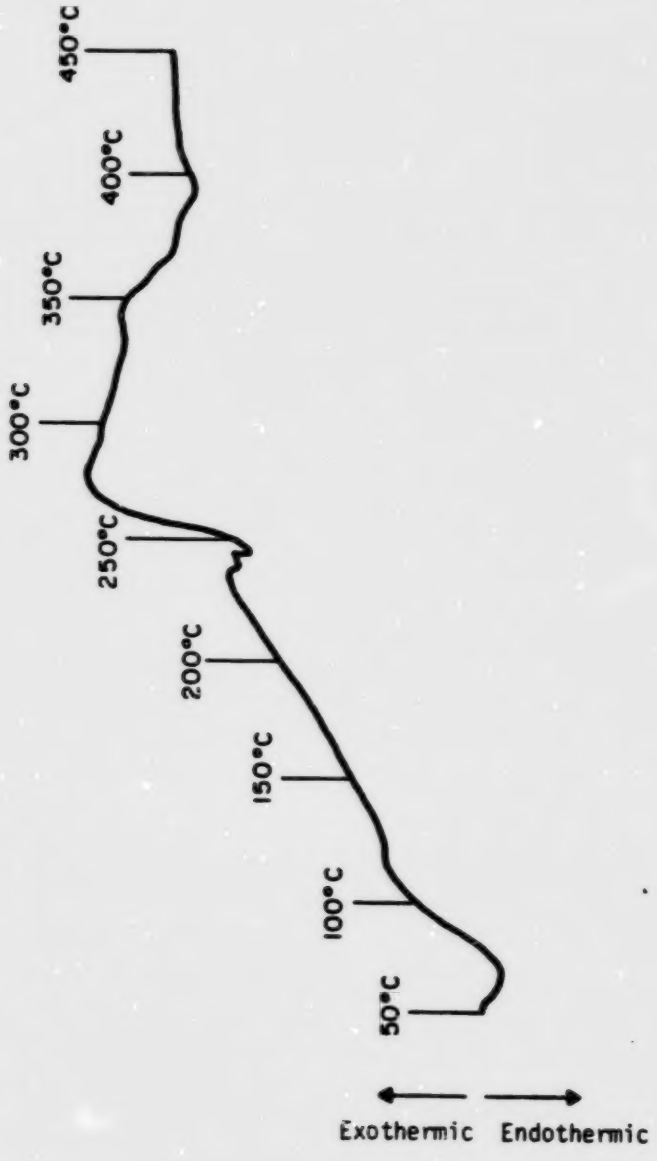
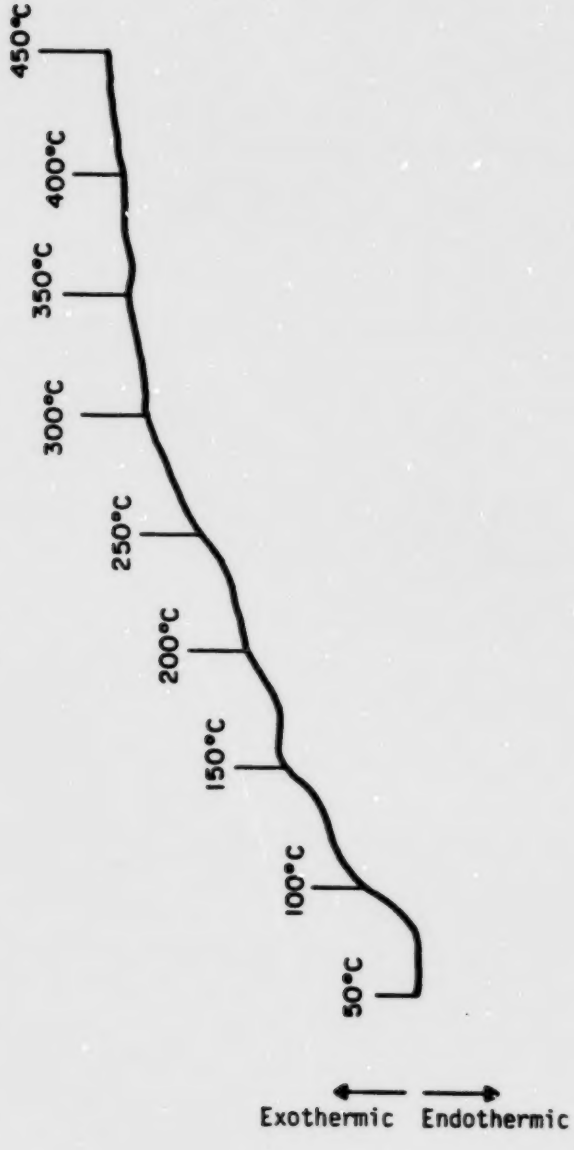


FIGURE 3.7

DTA THERMOGRAM OF HEAT-TREATED FIBRE



In order to assure complete removal of the adsorbed surfactant, the fibre was heat treated at 350°C for three hours. The subsequent differential thermogram (Figure 3.7) shows no significant features up to 450°C suggesting that the surfactant has indeed been removed. It is also noted that the majority of the unbound brucite observed in the dry-milled fibre has also been converted by this heat-treating process.

Surface area analysis of the "surfactant-free" samples by the B.E.T. method gave the following results:

Sample	B.E.T. (m^2/g)	Air Permeability (From Figure 3.1)
Ball milled 30 mins. in 0.1% OT	13.5	--
As above but washed in methanol	20.7	15,700
As above but heat treated	24.6	25,700

From these results, it appears that the surfactant does indeed bond the asbestos fibrils together to decrease the effective surface area. The conclusion, based on DTA, that the residual surfactant is removed by methanol washing and heat treating is further confirmed by the B.E.T. results which show heat treatment to be the most effective removal technique.

4. DRY MILLING

4.1 Description of Equipment

A batch process pilot plant facility designed to process bulk ore samples and large diameter (30 cm) drill cores currently exists at Ontario Research. The equipment housed in this facility are scaled-down versions of equipment used conventionally in asbestos dry-milling operations. A description of the equipment used in this evaluation is as follows.

4.1.1 Screens

A 76 cm x 152 cm Rotex Screener (Model 201) was used for all screening operations.

Material fed to the screener is first separated in the conventional screening manner into fractions that are either smaller or larger than the pre-selected screen cloth opening. As the oversize fraction travels down the screener, the material segregates into a bottom layer consisting predominately of rock fragments and a top layer consisting predominately of asbestos fibre. At the oversize discharge, the fibrous layer is separated from the larger rock fragments by aspiration.

4.1.2 Fibrizers

A two-row (61 cm diameter) vertical type fibrizer (manufactured by Fournier Steel Works) was used for all fibrizing operations. The number of hammers (4) and the hammer tip-speed (2,400 m./min.) also remained constant for all fibrizing operations.

Material fed to the fibrizer is impacted by the rotating hammers and stationary baffles. The resultant product from the impaction consists of a size reduction of rock fragments and opened fibre.

4.1.3. Cyclones

A 152 cm diameter cyclone was used to collect all the fibre products aspirated or 'lifted' from the various screening operations. A 30 cm diameter rotary dump valve affixed to the cyclone underflow was used to remove the various fibre products.

4.1.4 Dust Collector

A Wheelabrator (No. 72 Model 84) pulse-type dust collector was used for all process and hygienic dust removal operations.

4.2 Description of Asbestos Ore

A mill head sample of asbestos ore was received by Ontario Research from a Canadian producer. A visual inspection of this sample revealed that a considerable amount of the fibre appeared to be well liberated from the gangue. This fibre also appeared to be sufficiently 'opened' for conventional dry-processing techniques. A further comment on the 'degree of openness' of the fibre will be discussed Section 4.3.

4.3 Process Description and Procedure

One representative sample of ore was processed through a 'typical' dry-milling process which is used conventionally for processing exploratory drill cores for asbestos fibre evaluation. The flow sheet employed for this process is attached as Figure 4.1.

The ore sample was first screened on a 3-mesh (6.63 mm) screen cloth. The resultant fibre aspirated from this operation was designated as L-1. The oversize fraction was passed through the fibrizer to release and/or open any fibre bundles present.

The undersize and fiberized oversize fractions were then combined and screened on a 14 mesh (1.30 mm) screen cloth. The resultant fibre aspirated from this operation was designated as L-2. Both the oversize and undersize fractions were then fibrized to further open the fibre present.

The fibrized material was finally screened on a 35-mesh (0.51 mm) screen cloth. The aspirated fibre was designated as L-3. The undersize and oversize fractions were designated as T-1 and T-2, respectively.

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FIGURE 4.1

DRY MILLING FLOW SHEET

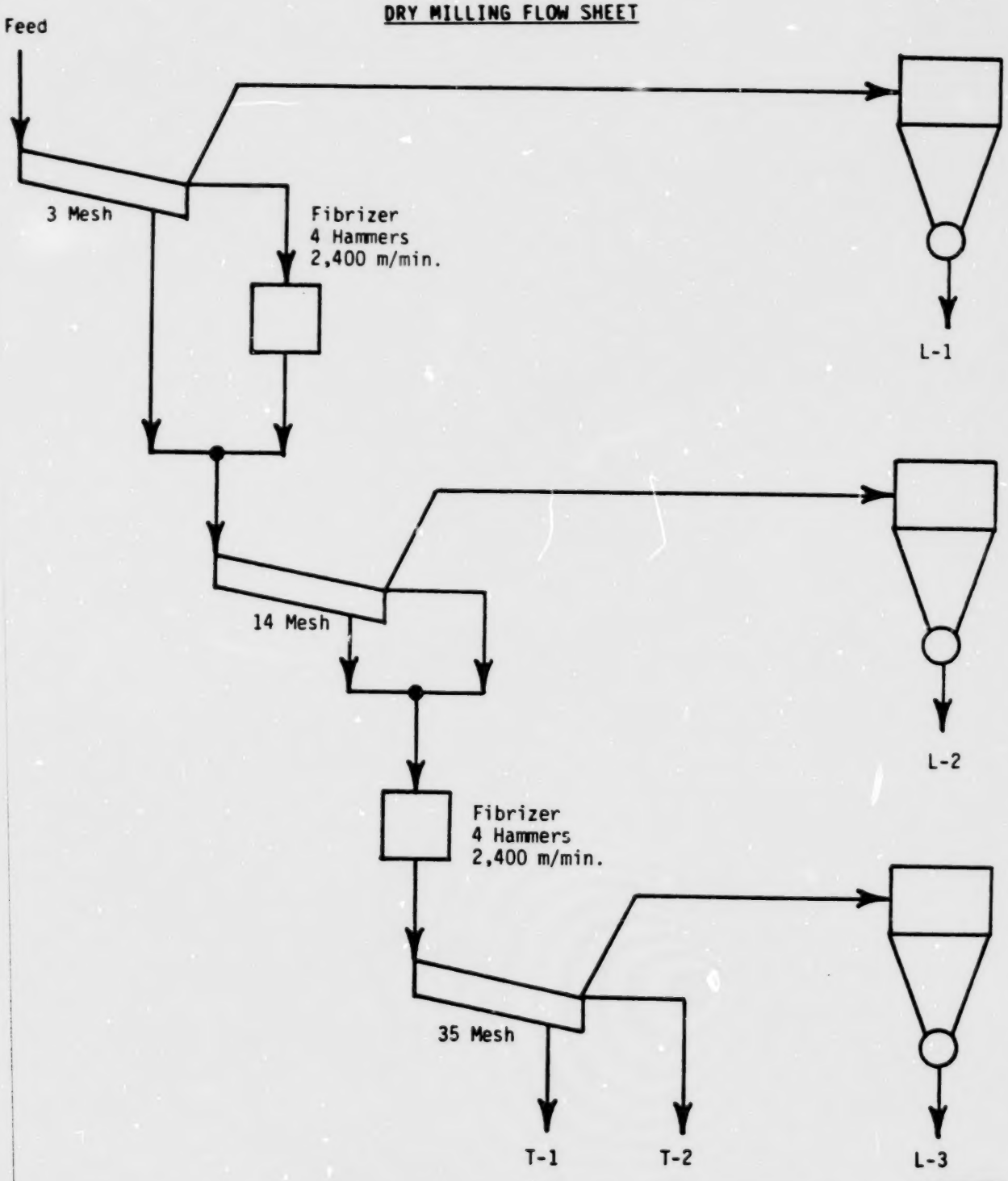


TABLE 4.1

ROCK AND FIBRE DISTRIBUTION FOR DRY MILLED FIBRE

Fraction	L-1 FIBRE LIFT			L-2 FIBRE LIFT			L-3 FIBRE LIFT		
	Wt. (kg)	Rec. (% Wt.)	Dist. (% Wt.)	Wt. (kg)	Rec. (% Wt.)	Dist. (% Wt.)	Wt. (kg)	Rec. (% Wt.)	Dist. (% Wt.)
Feed	231.8	100.0	100.0	225.7	97.4	100.0	207.6	89.6	100.0
Oversize	115.4	49.8	49.8	133.9	57.8	59.3	134.1	57.9	64.6
Undersize	114.1	49.2	49.2	75.6	32.6	33.5	62.1	26.8	29.9
Fibre	1.1	0.5	0.5	15.6	6.7	6.9	7.7	3.3	3.7
Loss	1.2	0.5	0.5	0.6	0.3	0.3	3.7	1.6	1.8

COMMENTS

(1) Recovery values are related to the initial head weight of the ore sample (231.8 kg).

(2) Distribution values are related to the feed weight of the fibre lift in question.

TABLE 4.2

MATERIAL BALANCE FOR DRY MILLED FIBRE

	FIBRE PRODUCTS				REJECTS				
	L-1	L-2	L-3	Total Fibre	+35	-35	Baghouse	Total	Loss
Wt. (kg)	1.1	15.6	7.7	24.4	134.1	62.1	4.3	224.9	6.9
Rec. (%)	0.5	6.7	3.3	10.5	57.9	26.8	1.8	97.0	3.0

All fractions collected from the various unit operations were weighed prior to further processing. The dust collector or 'Baghouse" product was also weighed for material balance purposes. The results of the dry milling test are shown in Tables 4.1 and 4.2.

5. WET COMMINUTION

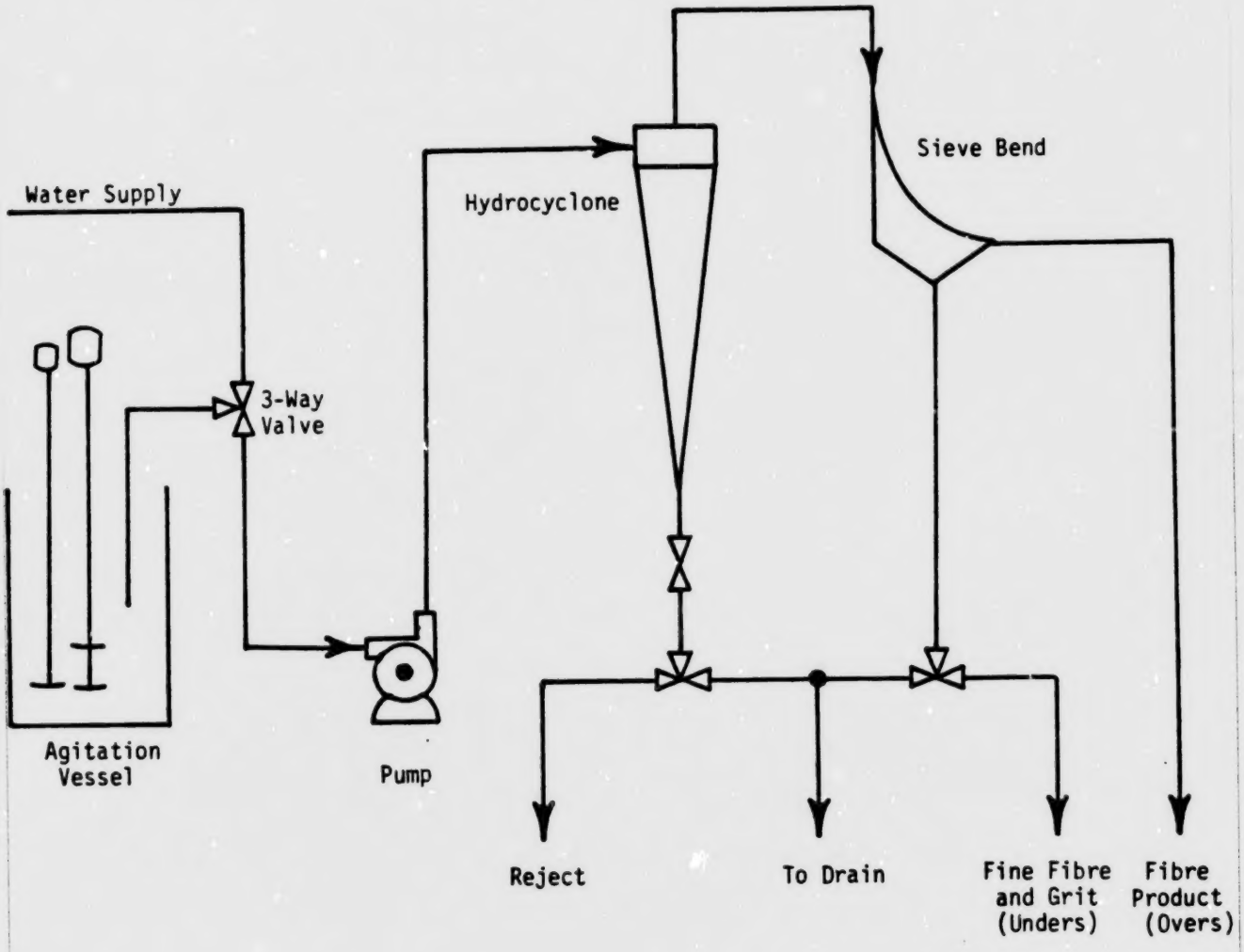
5.1 Preliminary Flow Sheet Design

Based upon the results and observations of the exploratory bench scale studies, a preliminary wet comminution flow sheet for ore was designed (a schematic is shown in Figure 5.1). An initial proving test was conducted using dry processed fibre tailings already on hand at Ontario Research. These tailings consisted of a rock top-size of 35 mesh (0.51 mm) and therefore, could easily be accommodated by the system.

A Centri-Cleaner (Model 600-3) hydrocyclone manufactured by Bauer Brothers was selected for this investigation. This model has been designed for fibre degritting in the pulp and paper industry. Since the hydrocyclone has the ability to separate particles of different size and shape, chrysotile asbestos fibres may be separated from its approximately equidimensional gangue material. The asbestos fibres and gangue material will therefore be discharged as overflow and underflow, respectively.

FIGURE 5.1

PRELIMINARY WET MILLING FLOW SHEET



The dilute fibrous overflow slurry was subsequently thickened on a sieve bend. A Hydrasieve (Model 522 - 18) sieve bend manufactured by Bauer Brothers was selected for this investigation. The headbox of the Hydrasieve was not utilized; instead, a slurry spreader was constructed and affixed above the top end of the screen. A preliminary test run with water only revealed that this set-up would satisfactorily spread the slurry across the width of the sieve. The screen utilized consisted of wire spacings of 0.762 mm.

The flow sheet was designed to be continuous from one unit operation to the other and, therefore, transportation requirements were minimized to one pump. However, in order to achieve steady state during the entire test run, water only was first passed through the system. When steady state was obtained, the slurry was then introduced to the system via a three-way valve arrangement shown in Figure 5.1. Water only was again passed through the system at the completion of the test run.

Excellent fibre/gangue separation was observed to occur in the hydrocyclone. The wire spacings of the sieve bend, however, were too large and caused in all of the hydrocyclone overflow to be reported in the sieve bend undersize fraction. This problem can be rectified by replacing the screen with one consisting of smaller wire spacings.

5.2 Process Description and Procedure

Based upon the encouraging results obtained from the tailing sample in the preliminary test run, a representative sample of the Canadian ore was processed. Although segregation of the larger rock fragments and fibre phases occurred in the agitation vessel, rock fragments large enough to plug the hydrocyclone were still present in the finer phase. In order to rectify this problem, the preliminary flow sheet design required some modifications (see Figure 5.2).

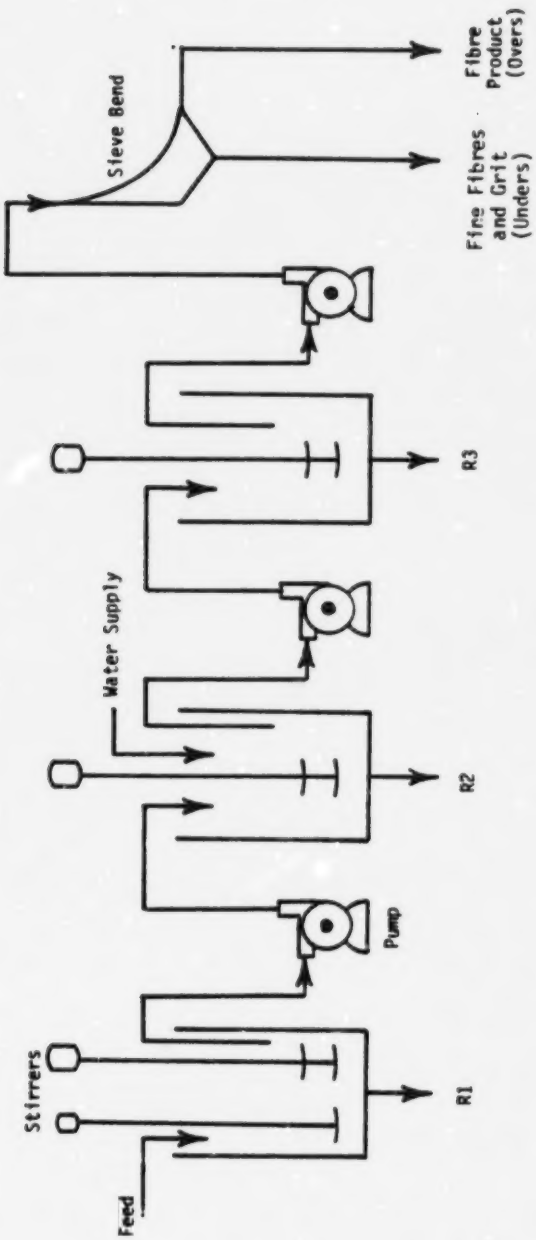
The hydrocyclone was replaced in the system with a series of slurry agitation and decantation stages. The conditioned ore was agitated in a 208-litre drum at approximately 10% solids by two mechanical stirrers operating at 1768 and 360 rpm, respectively. An additional impeller was fitted to the 360 rpm shaft approximately mid-drum. This motion was sufficient to effect a separation of fibre and grit from the larger gangue which remained near the bottom of the vessel. The fibre and fine grit from the top half of the drum was subsequently removed and collected in a second drum via an impeller type pump (75 l/min). Water was added to the original drum and the ore agitated again to induce more fibre to the top of the barrel. This fraction was once again pumped off into the collection barrel and when the level had dropped to a point at which larger rock fragments were being lifted into suspension, the high speed rotor was turned off. The same procedure was executed for the slow speed stirrer when the slurry composition necessitated (approximately 1/3 full). This allowed the heavier rock and grit to settle and thus, avoid being collected. The remaining fibre, grit and rock was dried and weighed as reject 1. (R1)

A second separation stage was performed on the fibre and grit collected in the first step. The procedure was executed as in the primary separation except only the slower speed rotor was used. The pumped slurry was collected in a third barrel and the remaining fibre and grit dried and weighed as reject 2 (R2).

The final separation involved pumping the contents of barrel 3 over the sieve bend to aid in the dewatering of the fibre product. The slow speed stirrer was used for approximately the first half of the slurry and gentle hand agitation used for the remainder. The remaining fibre and grit in barrel 3 was filtered, dried and weighed as reject 3 (R3). The unders from the sieve bend were also collected and yielded a composite of fine fibre and grit.

FIGURE 5.2

WET MILLING FLOW SHEET



Two batches of conditioned ore were processed in the manner described above:

(a) ball milled ore at 15% solids in water for 1/2 hour;

(b) ball milled ore at 15% solids in 0.7% Aerosol OT (based on mill feed) for 1/2 hour.

The aqueous ball milling was conducted as a control to examine the effects of using a surfactant.

The final fibre product from each of these runs was collected and used in subsequent product evaluation testing (Section 7). Based upon the observed length of the fibre product generated by using a ball milling technique, an experimental run consisting of ore pretreated by 1/2 hour mechanical stirring in water was also processed according to the flowsheet of Fig. 5.2. This test would be expected to "lift" only the free fibre present in the ore without introducing any severe mechanical action that may reduce fibre length.

In another attempt to employ the hydrocyclone as a wet separation technique, one test run was conducted on the -3 mesh fraction of the ore from the dry processing mill (see Figure 4.1). The -3 mesh fraction was first subjected to a fibrizing step (four hammers, 2400 m/min.) to aid in the size reduction of rock fragments and assist in fibre liberation. Although the hydrocyclone was very efficient in removing large grit from the fibre, a considerable amount of the fibre was lost with the hydrocyclone underflow. The experiment was discontinued at this stage until further optimization could be attained.

TABLE 5.1

MATERIAL DISTRIBUTION OF THE WET COMMINUTION TEST RUNS

Test Run	Head Weight (kg)	SIEVE BEND				REJECTS						LOSS	
		Fibre Product		Fine Fibre and Grit		R1		R2		R3			
		Wt. (kg)	Rec. %	Wt. (kg)	Rec. %	Wt. (kg)	Rec. %	Wt. (kg)	Rec. %	Wt. (kg)	Rec. %	Wt. (kg)	Rec. %
Aqueous Ball Milled	22.7	1.8	7.9	4.8	21.2	13.0	57.3	0.9	3.9	0.5	2.2	1.7	7.5
Chemical Ball Milled	22.7	2.9	12.8	3.4	14.9	13.8	60.8	1.2	5.5	0.4	1.6	1.0	4.4
Aqueous Stirred	24.5	1.1	4.5	1.4	5.9	20.5	83.7	0.6	2.4	0.2	0.6	0.7	2.9

6. FIBRE EVALUATION

In the following analysis, the composite dry milled sample represents fibre obtained from the three dry lifts.

6.1 X-ray Analysis

Four samples of asbestos were analyzed by X-ray fluorescence and X-ray diffraction. The samples were labelled as follows:

(1) Composite dry milled sample
(2) Ball milled one hour in 0.1% Aerosol-OT solution
(3) Ball milled one hour in 0.1% Aerosol-OT solution, then heated at 350°C for three hours
(4) Ball milled one hour in water

6.1.1 X-ray Fluorescence Analysis

Two samples of each material were prepared for X-ray fluorescence analysis. The first sample, for semi-quantitative X-ray fluorescence scan, was milled for 10 minutes in a Shatterbox using a tool steel vial. The milled product was pelletized at 25 tons in a 40 mm die. Results of

the semi-quantiative scans are shown in part (B) of Table 6.1. These results show a significant increase in the detectable sulphur concentration for the two samples treated with Aerosol OT, suggesting a possible adsorption of the sulphosuccinate compound.

The second sample, for quantitative X-ray fluorescence analysis of selected elements, was prepared by vacuum drying, and milling in a tungsten carbide vial in a vibratory mill. The milled material was fused in a borate glass and analyzed against reference standard fusions. Results are shown in part (A) of Table 6.1.

The increase in Al_2O_3 and SiO_2 concentration is believed to be a result of the ceramic ball mill degradation.

6.1.2 X-Ray Diffraction Analysis

X-ray diffraction scans were obtained from the pelletized samples described above. Mineralogical compositions are given in Table 6.2, along with estimates of the concentration of each phase.

The dry-milled material is mostly chrysotile, with small amounts of brucite, magnetite, hematite, and calcite. The wet-milled samples (2, 3, and 4) contain additional amounts of mullite, corundum and quartz most likely due to the physical degradation of the ball mill. The X-ray fluorescence results indicate higher concentrations of Al_2O_3 than can be accounted for in the X-ray diffraction scans. This probably means that some mullite and/or alumina is present in non-crystalline or finely-divided form.

The diffraction characteristics of the chrysotile mineral have not been seriously changed by the sample treatment, except for a reduction in overall diffraction intensity due to the introduction of additional phases in Samples 2, 3, and 4. Table 6.3 lists the (004) and (060) peak heights (in chart units) which illustrate this decrease. The (004) peak "half-width" (i.e. width at half height) is somewhat variable, but this could be influenced by grinding during sample preparation as well as by the sample treatment.

It is interesting to note that the small amount of unbound brucite present in the dry-milled fibre has been removed by heat treating at 350^{o}C for three hours. This further confirms the observations presented in Section 3.3.

TABLE 6.1

X-RAY FLUORESCENCE RESULTS

	Dry Milled	Chemical Ball Milled	Chemical Ball Milled & Heated	Aqueous Ball Milled
(A) Quantitative Analysis (Wt. %)				
SiO_2	39.53	41.87	42.91	41.74
Al_2O_3	0.63	16.95	17.68	11.53
Fe_2O_3	5.65	3.50	3.60	4.48
MgO	40.51	25.08	24.62	29.00
CaO	0.45	0.47	0.49	0.36
K_2O	0.01	1.40	1.43	0.92
Loss on Ignition	13.22	10.73	9.27	11.97
(B) Semi-quantitative Analysis (Approx. Wt. %)				
S	0.03	0.2	0.2	0.03
Cl	0.3	0.02	0.03	0.02
Ti	0.01	0.03	0.03	0.05
Cr	0.1	0.1	0.1	0.08
Mn	0.1	0.09	0.09	0.09
Co	0.005	0.005	0.005	0.005
Ni	0.05	0.04	0.04	0.04
Cu	0.005	0.02	0.02	0.01
Zn	0.002	0.005	0.005	0.003
Br	0.003	--	--	--
Sr	0.002	0.002	0.002	0.002
Rb	--	0.004	0.004	0.006
Zr	--	0.002	0.002	0.004

TABLE 6.2

X-RAY DIFFRACTION RESULTS (Approx. Wt. %)

	Dry-Milled	Chemical Ball Milled	Chemical Ball Milled & Heated	Aqueous Ball Milled
Chrysotile	balance	balance	balance	balance
Brucite	2	2	--	1
Magnetite	2	2	2	2
Hematite	1	1	1	1
Calcite	1	1	1	1
Mullite	--	5	5	5
Corundum	--	5	5	5
Quartz	--	1	1	1

TABLE 6.3

X-RAY DIFFRACTION PEAKS

	Dry Milled	Chemical Ball Milled	Ball Milled & Heated	Aqueous Ball Milled
(004) Peak Height	91	75	42	50
(004) Half-Width (2θ)	0.57°	0.57°	0.64°	0.60°
(060) Peak Height	15	12	12	12

- heights are in chart units (200 cps per unit)
- half-widths are in degrees (2θ)

6.2 Electron Optical Studies

Two samples of chrysotile were examined in the analytical electron microscope to determine if differences in chemical or structural characteristics had been introduced by the method of comminution. Sample 1 had been dry-milled, and Sample 2 had been opened by wet ball-milling with 0.1% Aerosol OT, followed by heating at 350°C for three hours to destroy any residual traces of the surfactant. A sample of the wet ball-milled material taken prior to the heating step was also examined to determine if the surfactant was detectable at this stage.

The samples were dispersed in double-distilled water, and these dispersion were filtered through 0.1 μm pore size Nuclepore polycarbonate filters. Specimen grids were prepared from the filters using the carbon-coated Nuclepore filter procedure[42].

Thirty fibres from each of Samples 1 and 2 were examined by transmission electron microscopy (TEM) techniques.

All of the fibres yielded selected area electron diffraction (SAED) patterns, indicating that the fibres in both samples were crystalline. There appeared to be no significant differences between the SAED patterns from the two samples. Each fibre was also examined using energy dispersive X-ray analysis (EDXA) to determine the composition. The data are summarized in Table 6.4; these are quoted in terms of X-ray peak area ratios relative to silicon. It can be seen that no statistically-valid differences between the two samples could be demonstrated for the magnesium and iron concentrations relative to silicon.

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EDXA measurements were also made to determine if residual traces of the surfactant could be detected on the fibres. The X-ray count on each fibre was acquired for about 15 minutes, in order to achieve an adequate detection level for the very small amounts of sulphur present. The results are shown in Table 6.5. The S/Si peak area ratios were somewhat variable, and a low level of sulphur was present even in the dry-milled material. The statistical significance of the higher sulphur contents of Samples 2 (wet ball-milled, heated to 350°C) and 3 (wet ball-milled, before heating) was tested at a significance level of 0.05. Because of the high variability of the results on Sample 2, it was not possible with these limited data to demonstrate that the sulphur concentration was higher than that in Sample 1. A more definitive conclusion could probably be made if a greater number of fibres were examined. However, the sulphur content of Sample 3 was demonstrated to be higher than that of Sample 1, at a significance level of 0.05.

Typical fibrils from Samples 1 and 2 were examined at high magnification to determine if the two methods of comminution introduced morphological differences. Figures 6.1 and 6.2 show TEM micrographs of a typical fibril from each sample at a magnification of 450,000. It can be seen that there were no obvious morphological differences between these fibrils.

6.3 Surface Chemistry and Adsorption Properties

A determination of parameters, representative for the electrical surface charge of the chryostile fibres, such as the electrophoretic mobility and zeta potential is useful for the following reasons:

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TABLE 6.4

ENERGY DISPERSIVE X-RAY EXAMINATION OF CHRYSOTILE FIBRES

Sample	Number of Fibres Examined	Elemental Peak Area Ratios					
		$\frac{Mg}{Si}$			$\frac{Fe}{Si}$		
		Mean	Standard Deviation	Range	Mean	Standard Deviation	Range
1. Dry-Milled	30	0.836	0.055	0.724 - 0.958	0.087	0.021	0.064 - 0.153
2. Wet-Milled and Heated to 350°C	30	0.818	0.063	0.712 - 0.934	0.079	0.028	0.040 - 0.179

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TABLE 6.5

RESULTS OF EDXA EXAMINATION OF FIBRES FOR PRESENCE OF SULPHUR

Sample	Number of Fibres Examined	Mean $\frac{S}{ST}$ Peak Area Ratio	Standard Deviation of Mean	At 5% Significance, do the data indicate that the sulphur content is higher than that of dry-milled chrysotile (Sample 1)?
1. Dry-Milled	5	0.0028	0.00064	-
2. Wet-Milled and Heated to 350°C	5	0.0043	0.0025	No
3. Wet-Milled (Before Heating)	5	0.0042	0.00082	Yes

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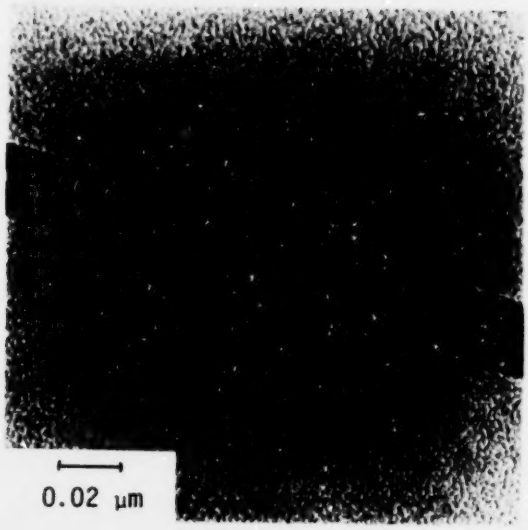

Figure 6.1 TEM micrograph showing typical fibril from dry-milled chrysotile.

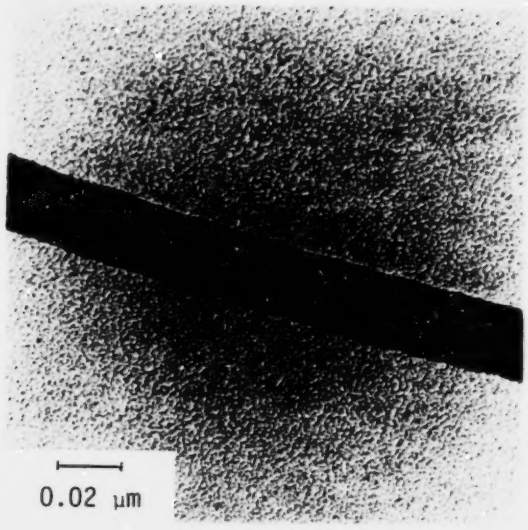

Figure 6.2 TEM micrograph showing typical fibril from chrysotile wet-milled with Aerosol OT, followed by heating to 350°C.

(a) It can give the surface charge of the untreated fibres over a wide pH range, including the pH range typical for the chemical comminution process.

(b) It establishes the type of surfactant that can be used and the useful pH range over which it can be applied.

(c) A comparison of the surface charges of the treated as well as natural fibres gives an indication of the chemical changes that have occurred during the comminution process.

Chrysotile asbestos fibre (empirical formula $Mg_3Si_2O_5(OH)_4$) has a structure consisting of alternating layers of silica and brucite ($Mg(OH)_2$). A structural misfit between these layers is believed to cause a curvature and thus, lead to the formation of distinct tubes (Figure 6.3). The aqueous chemistry of a typical chrysotile asbestos sample is shown in Figure 6.4. The suspension of asbestos fibrils has a positive surface charge in the pH range 5.5-10. The positive charge is the result of protonation or dehydration of surface (magnesium) hydroxyl groups. At pH values above 10, the hydroxyl groups are deprotonated to leave behind a negatively charged $Si-O-MgO^-$ group. At pH values below 5.5, Mg is removed to give uncharged Si-OH groups.

This model is consistent with two ZPC values, around pH 10 and 2, respectively. The ZPC value at lower pH is often not observed because of the slow rate of exchange between surface-Mg^{2+} and H^+(4).

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FIGURE 6.3 (43)

(A) Surface Structure of Chrysotile

(B) Cross-section of a Fibre Bundle

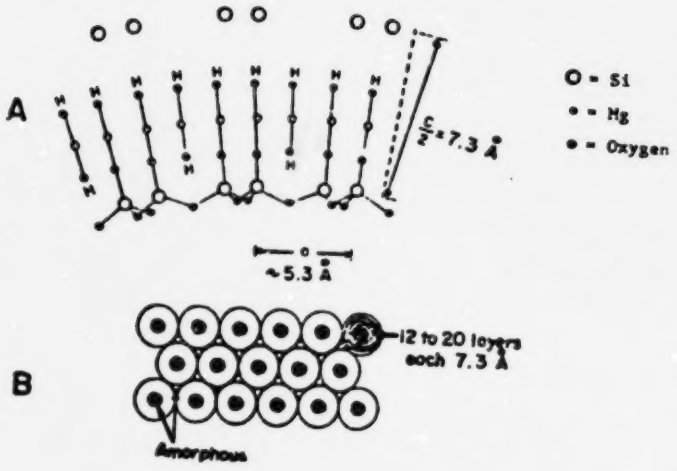
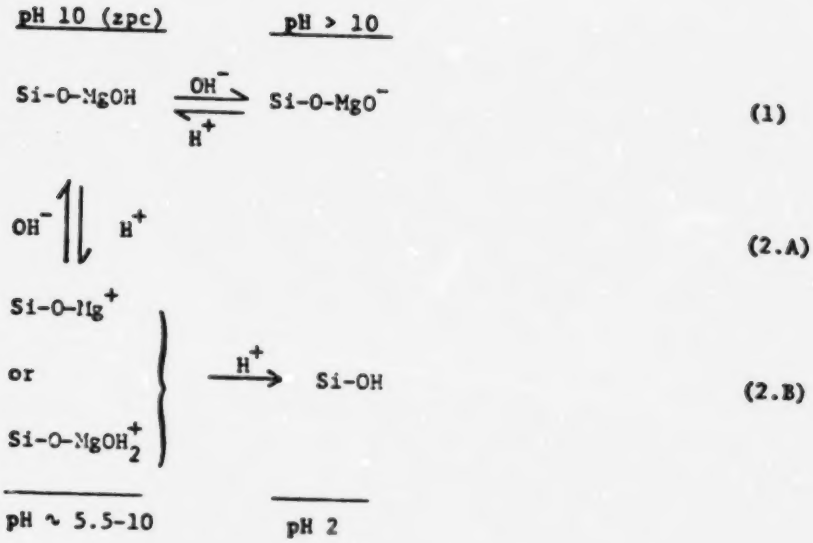


FIGURE 6.4

AQUEOUS CHEMISTRY OF CHRYSOTILE



6.3.1 Zeta Potential Measurements

To establish methodology a dry sample of a Quebec 7D-grade asbestos was used for zeta potential determinations on a Lazer-Zee meter. The zeta potential was measured in the pH range 3 to 10.

One gram of the sample was mixed with 500 mL of distilled water and dispersed in a Waring Blendor for about 60 seconds. This dilute slurry was then screened through a 325 mesh screen to obtain a filtrate of the desired particle size for the Lazer-Zee meter (50 μm or less).

The pH of this very dilute filtrate was then adjusted to 10 using 0.1 M KOH. A series of measurements of zeta potential was made and the average calculated. The Specific Conductance in mhos/cm was also measured on the Lazer-Zee meter.

Further adjustments to pH were made using 0.1 M HCl and 0.1 M KOH. At each pH point down to pH 3, zeta potentials and specific conductances were measured.

Table 6.6 gives the average measured results for the sample at each pH. The zeta potentials vs pH are shown in Figure 6.5.

TABLE 6.6

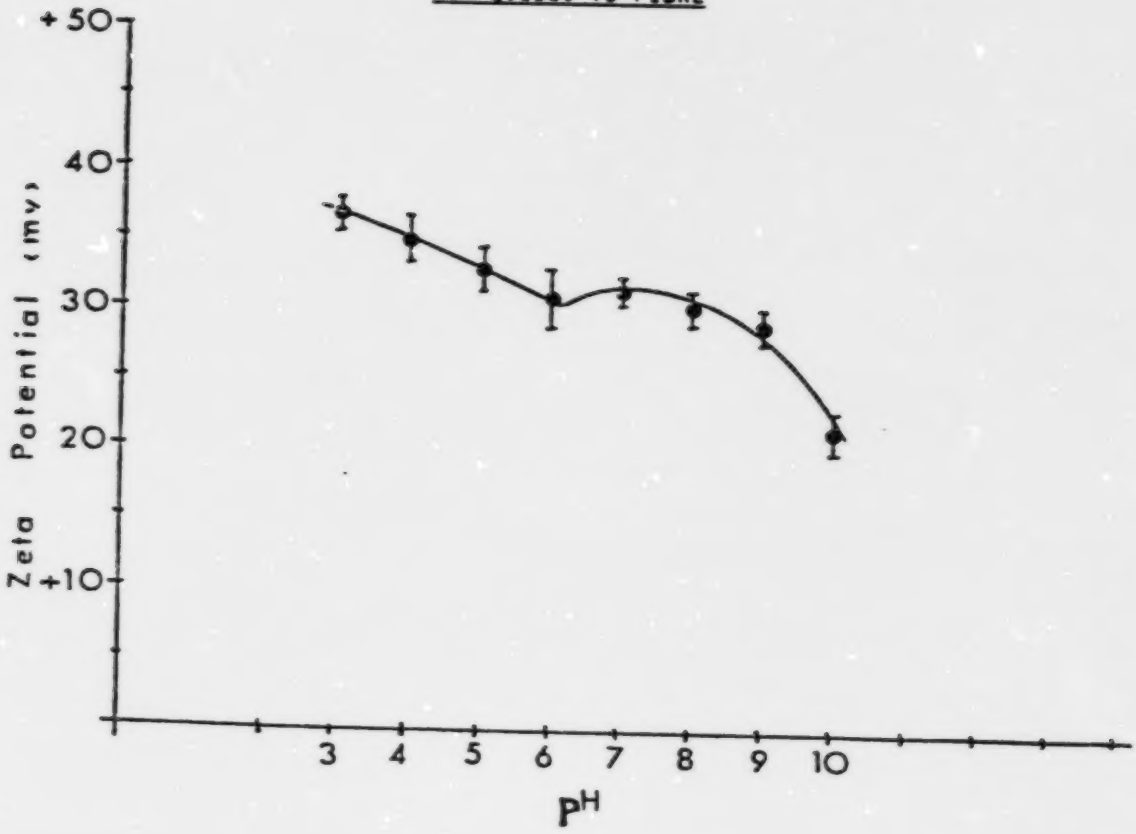
ZETA POTENTIAL AND SPECIFIC CONDUCTANCE AT pH 3 TO 10

	pH 3	pH 4	pH 5	pH 6	pH 7	pH 8	pH 9	pH 10
Zeta Potential (mev)	36.6 (σ=1.10)	34.9 (σ=1.45)	32.9 (σ=1.37)	30.8 (σ=2.09)	31.8 (σ=0.75)	30.2 (σ=0.73)	28.9 (σ=1.05)	20.8 (σ=1.60)
Specific Conductance (umhos/cm)	453	192	161	154	141	-	132	125

FIGURE 6.5

ZETA POTENTIAL SPECIFIC CONDUCTANCE AT pH 3 TO 10

FOR QUEBEC 7D FIBRE



The same procedure which was used for the Quebec asbestos sample was employed to produce the zeta potential vs pH curve of a fresh dry milled sample of the asbestos fibre under study. Upon slurrying and filtering through 325 mesh, a good particle cloud was obtained, and readings were made without difficulty at pH's 3 to 10. These results are presented on Table 6.7 and Figure 6.6.

With slurries of all the surfactant treated samples, it was observed that the fibre floats to the top in a thick and stable foam. This suggests that a substantial amount of the surfactant adheres to the fibres during processing. Initially, a chemically-comminuted sample (0.1% Aerosol OT surfactant) was prepared for zeta potential measurements. The filtrate of the slurry was crystal clear and no particles were visible on the Lazer-Zee. It was therefore suspected that the wet processing of the material and subsequent screening over the hydrasieve to remove water had also eliminated the fines and/or the fines were being "floated" and were present in the observed foam layer.

The same procedure was then attempted on a sample of the fibre obtained in the underflow of the hydrasieve. Some particles were produced after dispersion and screening through 325 mesh, although not enough to obtain reliable zeta potential readings. An attempt was made to increase the amount of small particles by dry ball milling for several hours, followed by slurrying and filtering.

The majority of fibre still tended to float at the top of the slurry in a foam but a considerable amount of fibre tended to agglomerate and settle out of suspension rapidly. The remaining suspended fibre was, however, sufficient to obtain Zeta potential measurements. These results are shown in Table 6.7 and Figure 6.7.

TABLE 6.7

ZETA POTENTIAL VS pH FOR A CANADIAN FIBRE

	pH 3	pH 4	pH 5	pH 6	pH 7	pH 8	pH 9	pH 10
Dry Milled Sample (Fig.1)	41.4 (σ=1.7)	43.1 (σ=1.7)	44.0 (σ=1.2)	45.1 (σ=1.3)	42.1 (σ=1.0)	41.6 (σ=1.5)	34.9 (σ=1.5)	28.0 (σ=1.3)
Chemically Comminuted, Sprayed with Surfactant (Fig.2)	+0.9 (σ=2.5)	-5.7 (σ=2.2)	-9.2 (σ=2.0	-12.1 (σ=1.9)	-8.1 (σ=2.4)	-8.5 (σ=3.4)	-9.3 (σ=3.6)	-8.0 (σ=2.7)

FIGURE 6.6
DRY MILLED SAMPLE

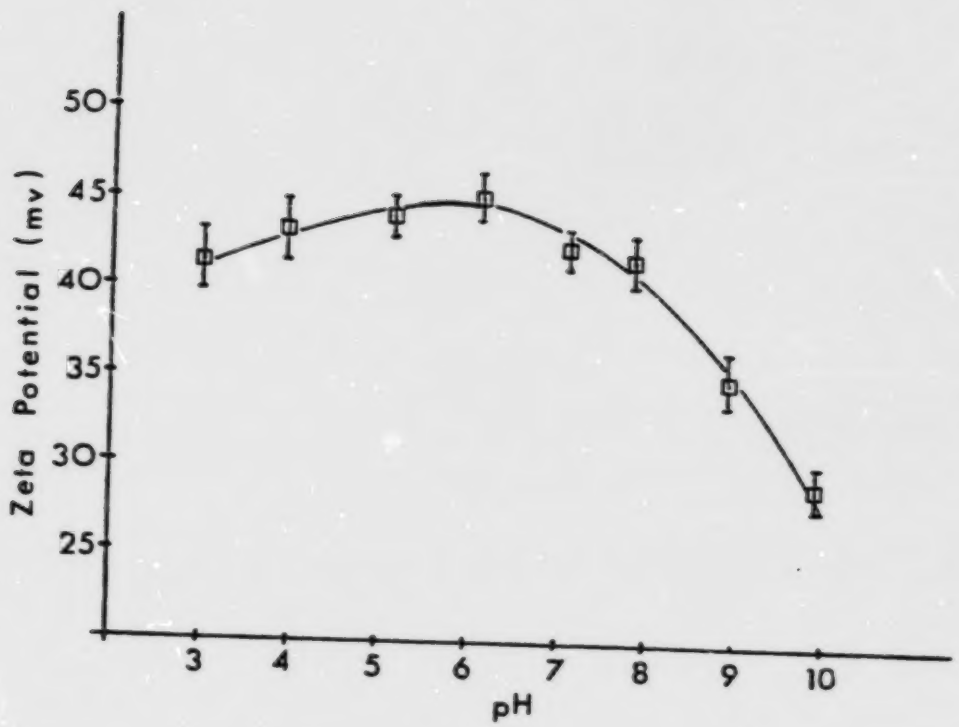


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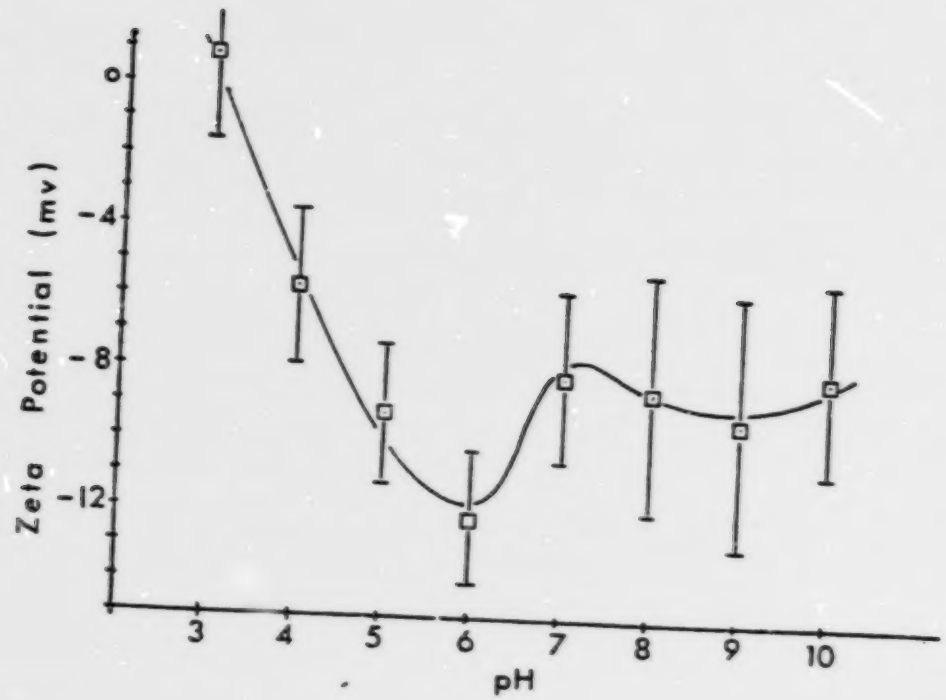
FIGURE 6.7
CHEMICALLY COMMINUTED SAMPLE
(with 0.1% Aerosol OT)



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7. PRODUCT EVALUATION

7.1 Introduction

Traditionally, the various fibre products of an asbestos mill are graded and utilized in accordance with their physical properties. Consequently, these characteristics are among the most important properties of the products. In Canada, the fibre products are generally tested according to methods established by the Quebec Asbestos Mining Association (QAMA).

The physical characteristics which are of interest to the asbestos industry are as follows:

- Degree of Fibrization
- Fibre Length Distribution
- Strength Imparting Properties
- Filtration Properties

Standard test procedures for measuring these physical characteristics of a fibre product are described in the "Chrysotile Asbestos Test Manual" and in the "American Society for Testing and Materials" test manuals. A brief description followed by a discussion of the results measured for each test procedure utilized for the current program is presented.

7.2 Degree of Fibrization

The degree of fibrization or subdivision of the asbestos fibre bundles in a sample can be measured by both direct and indirect methods.

The direct method utilized in the current program was the Turner and Newall Rapid Surface Area test (ASTM D 2752, QAMA D3). In this method, the resistance to airflow of a compressed sample of fixed mass and volume is measured. The resistance to airflow is directly dependent on surface area and is also directly related to the degree of fibrization which a given fibre sample has experienced. Thus, as the degree of fibrization increases, the surface area measurement also increases.

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The Turner and Newall Elutriation technique is another direct method of determining the degree of fibrization of a fibre sample. The primary components (crude, partially open, open and dust) of a fibre sample are separated from each other by successive stages of hydraulic elutriation. For a given fibre product, an increase in the degree of fibrization will decrease the amount of crude material present.

The degree of fibrization of a fibre sample may also be determined indirectly by the Wet Volume test method (QAMA D1). Briefly, a test sample is dispersed in water by means of a mechanical inverter and the resulting suspension is allowed to stand for a fixed period. The volume occupied by the fibre is measured by the solid-liquid interface. Generally, as the degree of fibrization increases, the volume occupied by the fibre also increases for a given fibre product.

A comparison of the degree of fibrization (measured by these three test procedures) for the fibre obtained in the following test processes are summarized in Table 7-1.

(A) Dry Milled
(B) Aqueous Ball Milled
(C) Chemical Ball Milled (0.1% Aerosol OT)
(D) Aqueous Stirred

The product obtained from the sieve bend unders in Runs B, C, and D were not evaluated individually, but due to the large extent of rock present, were disregarded.

Residual surfactant remaining on the surface of the fibre recovered from the chemically-treated test run (Sample C) caused difficulties in measuring the degree of fibrization by these test methods. The surfactant produced a hydrophobic fibre and consequently, the fibre would

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not remain dispersed in water while the tests were being conducted. The dispersion problem was particularly evident in the wet volume, elutriation and Bauer-McNett test. In all test procedures, the fibre tended to float in the test cells, thus inaccurate results were measured.

In order to rectify the dispersion problem caused by the surfactant, a representative portion of Sample C was heated for three hours at a temperature of 350°C. Although the colour of the fibre sample changed from white to brown, the dispersion problem was minimized. The results measured from the heated sample are discussed and compared with the other test runs in the remainder of this discussion.

The wet processed fibre products appear to be more open than the dry fibre product, as measured by both the rapid surface area and the wet volume test methods.

The results measured for the two ball mill fibre products, Sample B and C, appear to be very similar. The discrepancy between the "crude" and the "open" contents for these two fibre products could possibly be due to fibre adhesion caused by the heat treating of the fibre to remove the surfactant.

7.3 Fibre Length Distribution

The length distribution of an asbestos product can be measured by both wet and dry methods. The Bauer-McNett Wet Classification technique (ASTM D 2589, QAMA C1) is considered by the asbestos industry to be the single most reliable method of length distribution measurement. Basically, the Bauer-McNett Classifier fractionates a fibre sample by a wet screening technique. The relative amount of material retained on each screen is then compared to grade specifications which estimate fibre quality.

TABLE 7-1

COMPARISON OF THE DEGREE OF FIBRIZATION FOR THE DRY AND WET MILLED FIBRE PRODUCTS

Sample	Surface Area (cm^2/g)	Wet Volume* (ml)	Turner & Newall Elutriation (% Wt.)			
			Crude	Partly Open	Open	-200
Dry Milled	6,000	665	13.6	14.0	46.4	26.0
Aqueous Ball Milled	7,900	1,150	18.0	4.0	54.4	23.6
Chemical Ball Milled & Heated	7,500	1,090	29.6	5.2	41.6	23.6
Aqueous Stirred	9,100	1,980	22.4	3.2	59.6	14.8

*Average of two measurements

A comparison of the Bauer-McNett Classification results between the dry and wet milled fibre products is summarized in Table 7.2. These results indicate that the fibre recovered from aqueous stirring (test run D) is longer and cleaner than the fibre recovered from the other processing circuits including the conventional dry processing circuit. This is obviously due to the fact that only free fibre is recovered.

The results also appear to indicate that the fibre length (+4 and +14 mesh fractions of the classifier) is reduced by using ball milling at the retention times employed (Samples B and C). A rod mill coupled with a shorter milling time could possibly preserve the fibre length of the recoverable product.

7.4 Strength Imparting Properties

The Relative Strength test (ASTM D 3752) provides an estimate of the ability of asbestos fibres used to reinforce various asbestos-cement products. Briefly, asbestos-cement plaques are prepared with a standard quantity of an asbestos sample opened to a standard surface area. The modulus of rupture adjusted to a standard density of the asbestos-cement plaque is then compared to the specifications of a grading system.

A comparison of the asbestos-cement properties between dry and wet milled fibre products is summarized in Table 7.3. The observed strength may be directly related to the degree of long fibre present as indicated by the Bauer-McNett results.

7.5 Filtration Properties

The dewatering characteristics of an asbestos product are important to an asbestos-cement manufacturer. Two techniques to measure these characteristics were utilized in this program.

TABLE 7-2

COMPARISON OF THE FIBRE LENGTH DISTRIBUTION FOR THE DRY AND WET MILLED FIBRE PRODUCTS

Sample	Bauer-McNett Classification (% Wt.)*					
	+4	+14	Acc. +14	+35	+200	-200
Dry Milled	23.3	11.2	34.5	8.9	11.4	45.2
Aqueous Ball Milled	11.9	12.9	24.8	13.1	20.0	42.1
Chemical Ball Milled Heated	10.8	13.9	24.7	11.6	13.7	50.0
Aqueous Stirred	34.3	17.6	51.9	11.2	12.0	24.9

*Average of two measurements

TABLE 7-3

COMPARISON OF FILTRATION AND STRENGTH PROPERTIES FOR THE DRY AND WET MILLED FIBRE PRODUCTS

Sample	Freeness* (seconds)	Asbestos-Cement Properties			
		Surface Area** (cm^2/g)	Drainage Rate (ml/s)	Water Retention (% Wt.)	Modulus of Rupture (kg/cm^2)
Dry Milled	68	11,900	7.4	23.1	293
Aqueous Ball Milled	222	11.900	5.0	24.0	250
Chemical Ball Milled & Heated	431	12,200	4.4	23.9	193
Aqueous Stirred	85	11,850	6.5	23.5	341

* Measured prior to opening for the asbestos-cement measurements

** The surface area of the fibre used for the asbestos-cement measurements

In the freeness test (QAMA D7), a slurry containing the test sample and lime-gypsum water is thoroughly mixed in a mechanical inverter and dewatered in a specified filter. The time required for a fixed volume of the slurry to drain is measured. This technique is also affected by the degree of fibrization that the test sample has experienced.

The drainage rate of an asbestos-cement slurry is also measured during the Relative Strength test (ASTM D3752) discussed earlier. In preparing the plaques for the Relative Strength test, the rate (mL/s) with which the lime-gypsum water is drained from the asbestos-cement slurry is measured. Since the asbestos sample is opened to a specified surface area (see the Relative Strength technique), the drainage rate from one test sample to another can be accurately compared.

The filtration results (measured by these two test methods) for the fibre recovered in each test mill circuit are again summarized in Table 7-3.

Both the freeness and the drainage rate test methods indicate that the fibre recovered from the dry mill dewaters at a higher rate than the fibre recovered from any of the wet mill circuits tested. The two fibre products recovered from the ball mill circuits (Samples B and C,) appear to have significantly lower dewatering characteristics than those measured for the dry milled fibre product. This behaviour requires further study.

8. ENERGY ASSESSMENT

The electrical power consumption and throughput rates for each of the dry and wet ball milling process runs were measured. A Wattmeter was used to measure the electrical power requirement for each piece of machinery under load. In order to minimize any variations of the power consumption during the operation of a particular machine, the rate of material fed to the machine (throughput rate) was kept constant as practically possible. The average electrical power requirement for each piece of machinery was then recorded.

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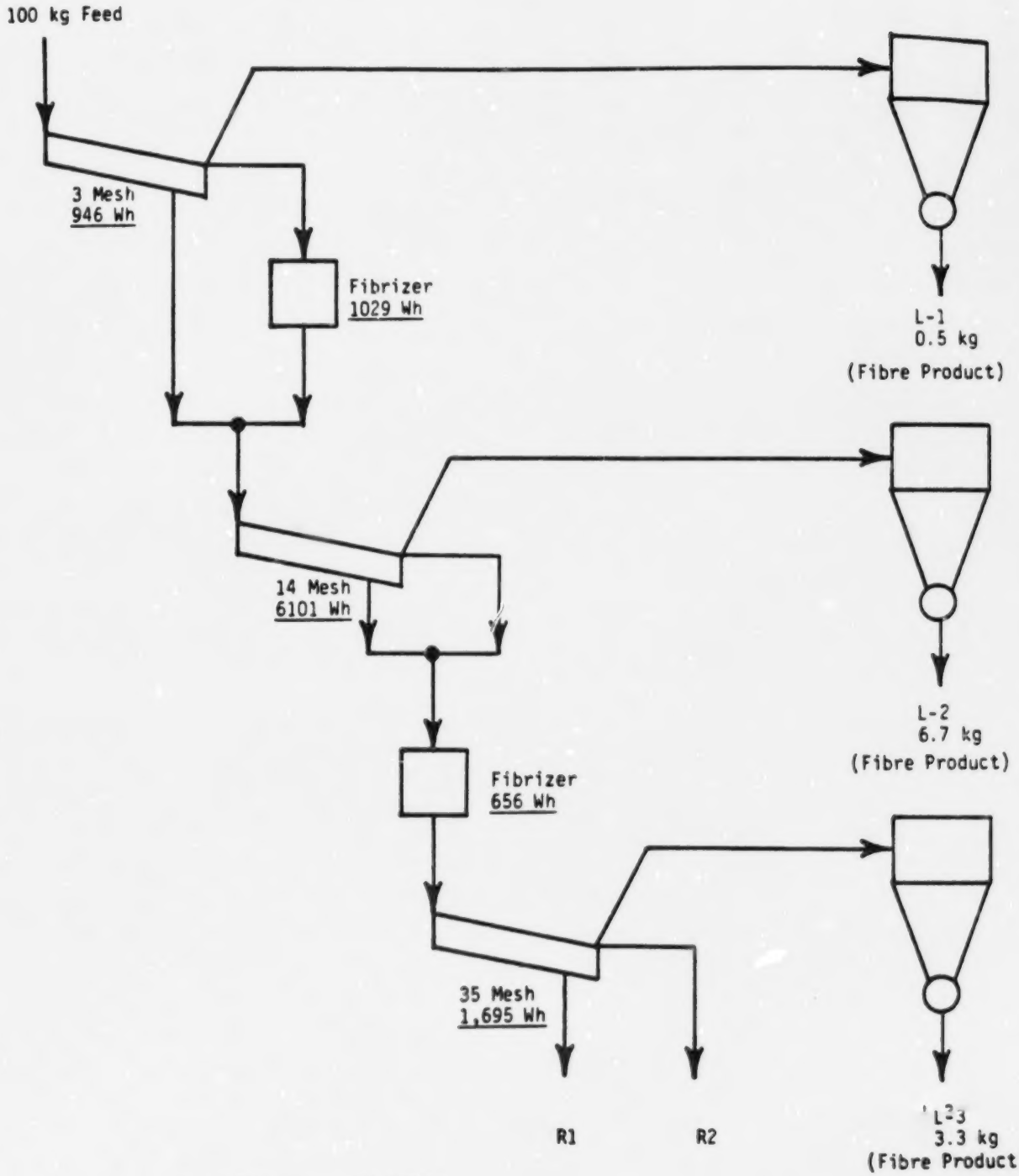
With the exception of the Baghouse, all of the equipment was measured for power consumption. Due to the present wiring arrangement of the Baghouse, installation of the Wattmeter was not possible.

Based on the run time and power requirements for each machine under load, the power consumption was determined for each unit operation in the various flow sheets tested. The total power consumption per kilogram of product recovered was then determined for each flow sheet (see Figures 8.1 to 8.3 for details). The power required to recover one kilogram of product from each flow sheet is summarized in Table 8.1.

These results clearly indicate that recovering fibre from a dry circuit is more energy intensive than recovering fibre from a wet circuit.

FIGURE 8.1

ENERGY CONSUMPTION FOR DRY MILLED FIBRE



Total Energy = 10,427 Wh

= 993 Wh/kg Fibre Product

FIGURE 8.2

ENERGY CONSUMPTION FOR THE AQUEOUS BALL MILLED FIBRE

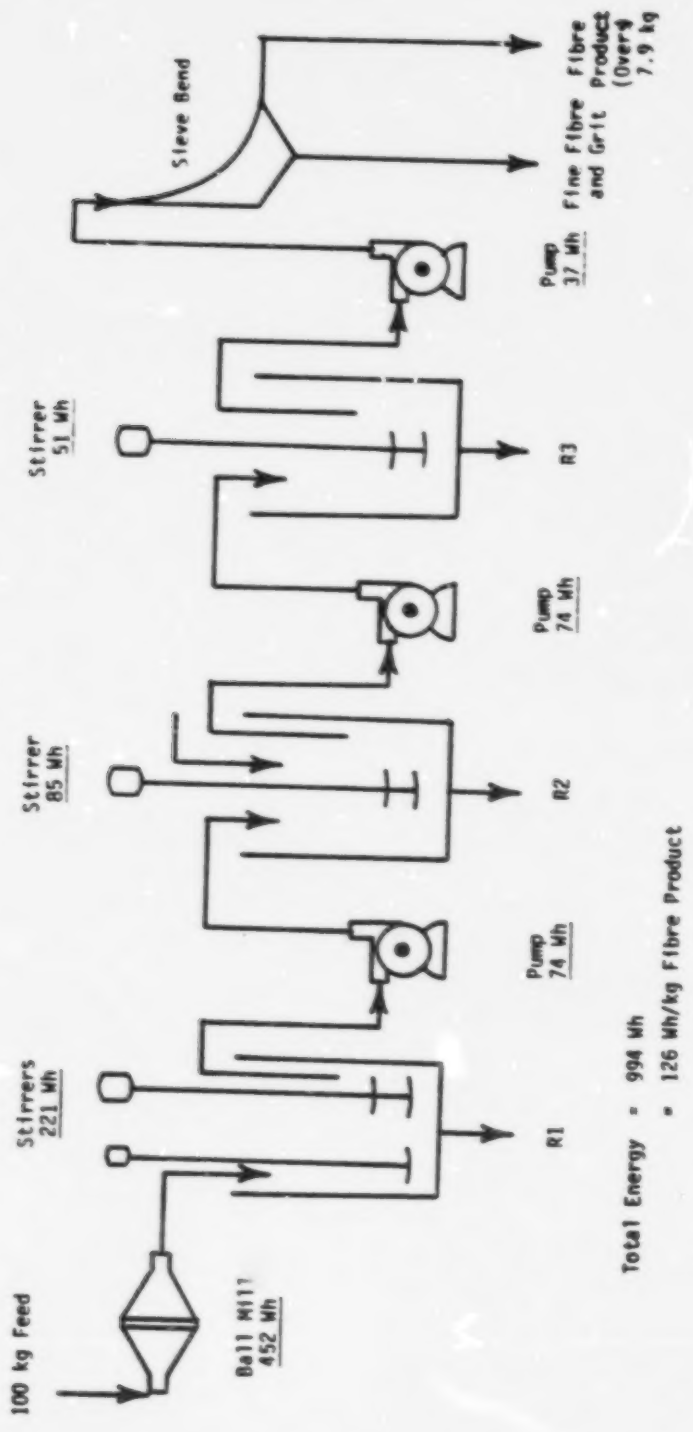


FIGURE 8.3

ENERGY CONSUMPTION FOR THE CHEMICALLY BALL MILLED FIBRE

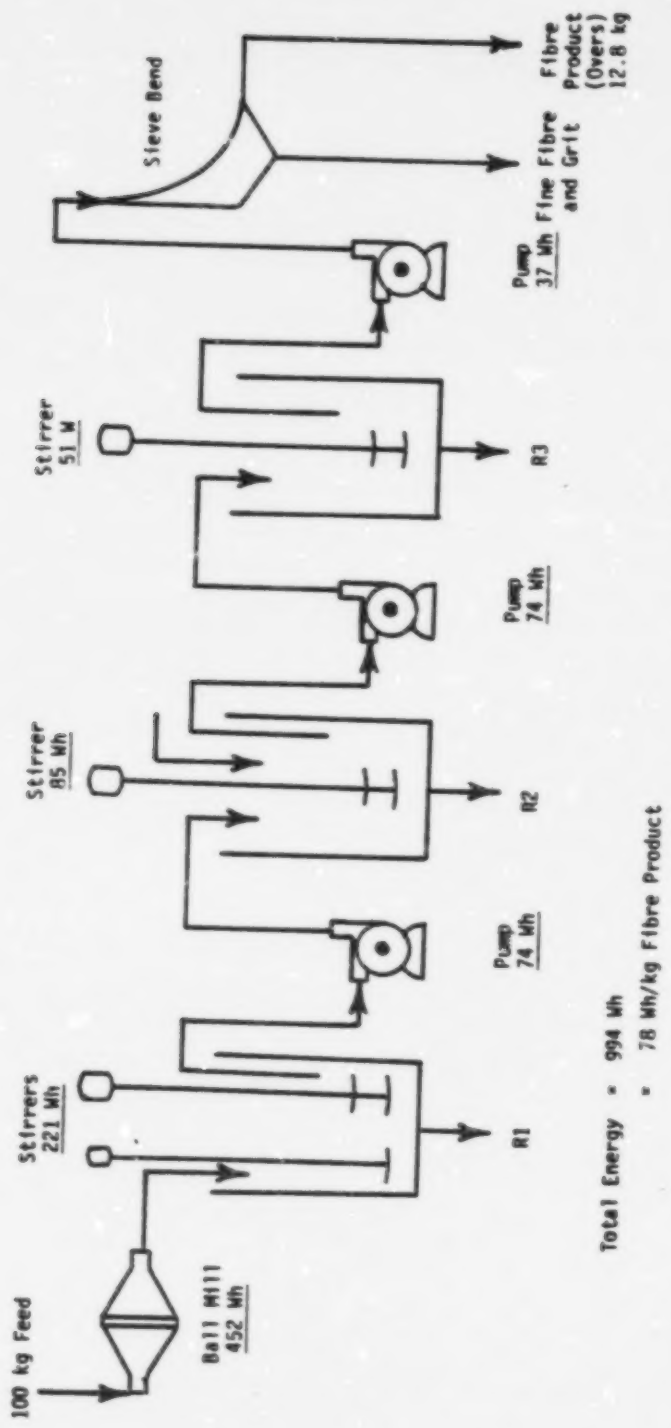


TABLE 8.1

SUMMARY OF THE POWER REQUIREMENTS FOR EACH PROCESS

Process	Power Required per 100 kg of Feed (Wh)	Total Fibre Recovery (%)	Power Required (Wh/Kg Product)
Dry Milling	10,427	10.5	993
Aqueous Ball Mill	994	7.9	126
Chemical Ball Mill	994	12.8	78

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9. DISCUSSION

In examining the practicality of employing chemical surfactants to aid in the recovery of asbestos fibre from its ore, it is important to realize that a proposed process may be very ore specific (i.e. may have to be adjusted to the type of ore). In the current study, the Canadian ore provided was free-milling and a considerable amount of fibre was already liberated from the gangue. This condition is not considered typical for all types of ore and certain changes may be required when processing a specific ore.

Preliminary bench scale studies examined the effect of surfactant concentration for both Aerosol OT and sodium oleate. It was observed in both cases that a high concentration of surfactant (0.5%) produced an unmanageable coagulated slurry which was extremely difficult to dewater and imparted a certain degree of stiffness in the dried fibre mats. For this reason, the large scale chemical processing was conducted at a lower surfactant concentration (~0.1%) to alleviate these problems.

One of the major problems encountered in using a chemical surfactant for the wet processing of the asbestos fibre, was the difficulty in characterizing the treated fibre. Previous studies [38, 39] have stated that up to 20% w/w of surfactant may be adsorbed on, or absorbed by, the asbestos fibre. The presence of residual surfactant was evident in this study through a number of qualitative and quantitative tests.

The differential thermal analysis (DTA) reported in Section 3 shows a large peak characteristic of an organic compound which may be indicative of the Aerosol OT used in treating the fibre. Electron optical studies (Section 6.2) also show a significant difference in the amount of elemental sulphur present on the chemically-comminuted fibre. This is most likely attributable to the adsorbed/absorbed sulphosuccinate component of Aerosol OT.

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The surface chemistry study of the chemically comminuted fibre (Section 6.3) indicates the presence of a negatively charged species on the fibre surface. Since this is not characteristic of natural asbestos fibre which has been shown to display a positive surface charge over the pH range examined, it is obvious that the surfactant has been bound to the fibre surface, thus rendering the fibres hydrophobic.

In all the test methods that required the fibre to be measured in an aqueous slurry form, problems with sample dispersion and foam generation were observed. In all cases, portions of the fibre sample would float on the water surfaces. This was particularly noticeable with the shorter fibre elements which were usually carried up with the foam layer.

From the difficulties encountered and based on the chemical assessment of the treated fibre, it was proposed to attempt some degree of surfactant removal. In Section 3, two types of surfactant removal techniques were addressed, heat treating and solvent/alkali washing. Owing to the relative ease and increased efficiency, the heat treating method was selected to provide "surfactant-free" chemically comminuted fibre for further qualitative/quantitive tests. This heat treating at 350^{o}C for three hours did not appear to degrade the fibre's crystal structure as measured using X-ray diffraction and electron optics (Tables 6.2 and 6.4).

The difficulties encountered in characterizing the chemically treated fibre by conventional test methods were not evident after the surfactant was removed. The fibre would readily disperse in water and foam generation was not observed. As a consequence, the heat treated fibre could readily be characterized by conventional test methods.

Australian practise [38, 39, 40] has been to contact/fibrize the fibre at more than 0.01% w/w and preferably more than 1% w/w surfactant (based upon mill feed). After fibrizing, the slurry is coagulated by excess dilution which decreases the surfactant concentration to between 0.01-0.1% w/w. In the current study, the fibre was ball milled at approximately 0.7% w/w (based upon mill feed), however the initial surfactant concentration was only reduced by approximately 50% in the subsequent agitation and decantation steps. This higher concentration (as compared to the Australian studies) is, therefore, the probable source of the difficulties encountered in qualitative and quantitative testing.

Reference to Table 9.1 clearly indicates that in this study the wet processing of asbestos ore using a ball milling technique and a surfactant significantly enhanced the recovered fibre yield over the conventional dry process (~12.8% vs 10.5%) while providing a significant reduction in energy consumption (78 Wh/kg vs 993 Wh/kg). This increased product yield is believed to be a result of a more gentle opening of the fibre bundles that would otherwise be damaged by mechanical action (i.e. fibrizing).

The low yield encountered in the aqueous ball milling run (7.9%) can be made comparable to the yields observed in the dry milling process if one considers the fibre that is present in the three rejects (see R1, R2, R3 of Fig. 5.2). Since these agitation/decantation stages were not optimized, a considerable amount of fibre was observed with the rock in the bottom of each vessel. Subsequent drying and fibre removal by Rotex aspiration revealed approximately 3.1% fibre (based on head weight) present in the rejects. This would increase the observed yields for the chemical and aqueous ball milling processes to 15.9% and 11.0%, respectively. Due to the preliminary nature of this study, however, no characterization tests were conducted on this recovered fibre, therefore, no comments can be made about its overall quality.

ORE

When comparing the differences in the energy consumptions of the various milling techniques employed in this study, it is important to note that:

(1) this study was considered to be preliminary in nature and the systems used were not optimized for maximum benefit (e.g. fibre quality or recovery);

(2) equipment selection was based on existing set-ups at ORF and direct correlation with industrial equipment was beyond the scope of the program;

(3) the energy data generated in this study relates to the fibre liberation from the rock and any pre-treatment of the ore or post-treatment of the fibre was not included (i.e. drying of ore, additional fibrizing of product, dewatering of fibre).

Nevertheless, the energy savings observed were real for the systems used and recoveries obtained, and can be taken as an indication that significant benefits would accrue if wet processing were used to liberate the fibre from the ore. It is also interesting to note that the energy consumed by the dry process techniques employed in this study (~1 Kwhr/kg) is of the same order of magnitude as that recently observed in an industrial mill[44] (2-3 Kwhr/kg) and also the previously referenced 1978 average energy figures quoted for the industry (~3 Kwhr/kg).

TABLE 9.1

SUMMARY OF ENERGY CONSUMED AND FIBRE QUANTITY RECOVERED FROM EACH PROCESS

Process	Energy Consumed (Wh/Kg Product)	Total Fibre Product (% Yield)
Dry Process	993	10.5
Aqueous Ball Mill	126	7.9
Chemical Ball Mill	78	12.8

10. CONCLUSIONS

The following conclusions are a result of assessing the work conducted in this study:

(1) Significant energy savings are observed for wet comminution of asbestos as compared to dry comminution.

(2) Fibre yield is increased significantly by employing ballmilling with a surfactant.

(3) Ball milling tends to shorten fibre (i.e. reduce fibre length).

(4) The use of surface active agents may have a detrimental effect on fibre properties.

(5) Multiple fibre grades may be produced by wet screening or classification.

11. RECOMMENDATIONS

Based upon the information and observations conducted in this project, the following recommendations for further study in the area of wet/chemical processing of asbestos, are proposed.

(i) Asbestos Ore Specificity

The liberation characteristics of asbestos fibre from different ore bodies may differ significantly, as can the individual fibre characteristics/properties. It is considered that alternate fibre types may require different surfactants and/or modifications to various unit operations in a wet processing flow sheet.

(ii) Effects of Milling on Fibre Quality

It is necessary in any asbestos milling operation to preserve fibre length and therefore the resulting high fibre value. In wet processing, it appears evident that the choice of the milling technique is critical to the preservation of fibre length, whether this be simple agitation, ball milling, rod milling or some other method. For this reason, a comparison and optimization of different milling techniques upon the fibre quality is required.

(iii) Survey of Available Surfactants

A wide variety of possible surfactants for use in the chemical comminution/dispersion of asbestos fibres have been suggested (29, 38, 39, 40). Criteria for the selection of a suitable chemical should include the degree of opening attained, the ease of handling, removal and recovery, as well as the availability, cost, and toxicity. The selection of a unique surfactant may also be very ore specific, and depend upon the surface characteristics of each particular fibre type. It is therefore proposed that a program to evaluate the above variables be implemented.

(iv) Effects of Chemical Treatment on Fibre Quality

The application of asbestos fibre to asbestos-containing products is highly dependent upon the surface properties of the fibre. The presence of absorbed/adsorbed organics resulting from the surfactant treatment may dramatically affect the observed characteristics of the final product (e.g. bonding at the fibre/matrix interface in asbestos cement products). If chemical comminution is to be used, it is necessary to determine the effects of adsorbed/ absorbed surfactants on the properties of end products and the tolerance levels which are acceptable.

(v) Preparation of Multiple Fibre Grades

Work to date indicates that possible fibre classification may be accomplished during the primary dewatering stage on the sieve bend. By proper selection of the screen opening of the sieve bend and/or the use of multiple units, several grades of fibre product may be recovered instead of a single composite. This aspect would most likely improve the marketability of the wet processed fibre product.

(vi) Combined Dry and Wet Processing

It is a necessity of the wet process that the ore be physically reduced (i.e. dry crushed) to a suitable size for pulping. Since long asbestos fibre is a premium product and is liberated at this stage, it is logical that the removal of the long fibre be effected at this time. In addition, this may be an opportune time to reject fibre-barren rock by standard scalping techniques. The subsequent beneficiated ore should then be pulped and evaluated for wet processing. Studies to examine this "front-end" classification should be initiated.

(vii) Marketability

In order to realize the significant energy savings of wet processing asbestos fibre, it is necessary to eliminate total drying of the recovered fibre and market the fibre product in a moist state (15 - 20% moisture), presumably as a felt. To assess the impact of this "novel" product on the asbestos fibre market, a feasibility study should examine the advantages and disadvantages of packaging, transportation, handling, and implementing a high moisture content fibre product.

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